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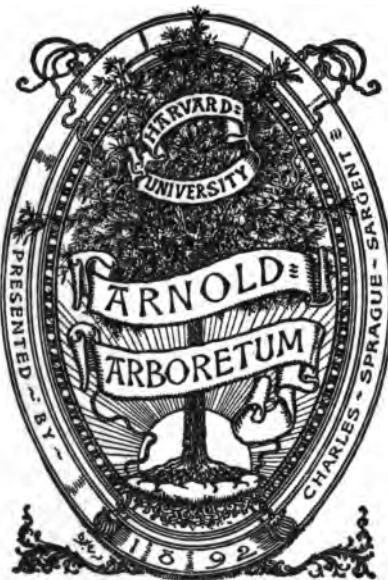
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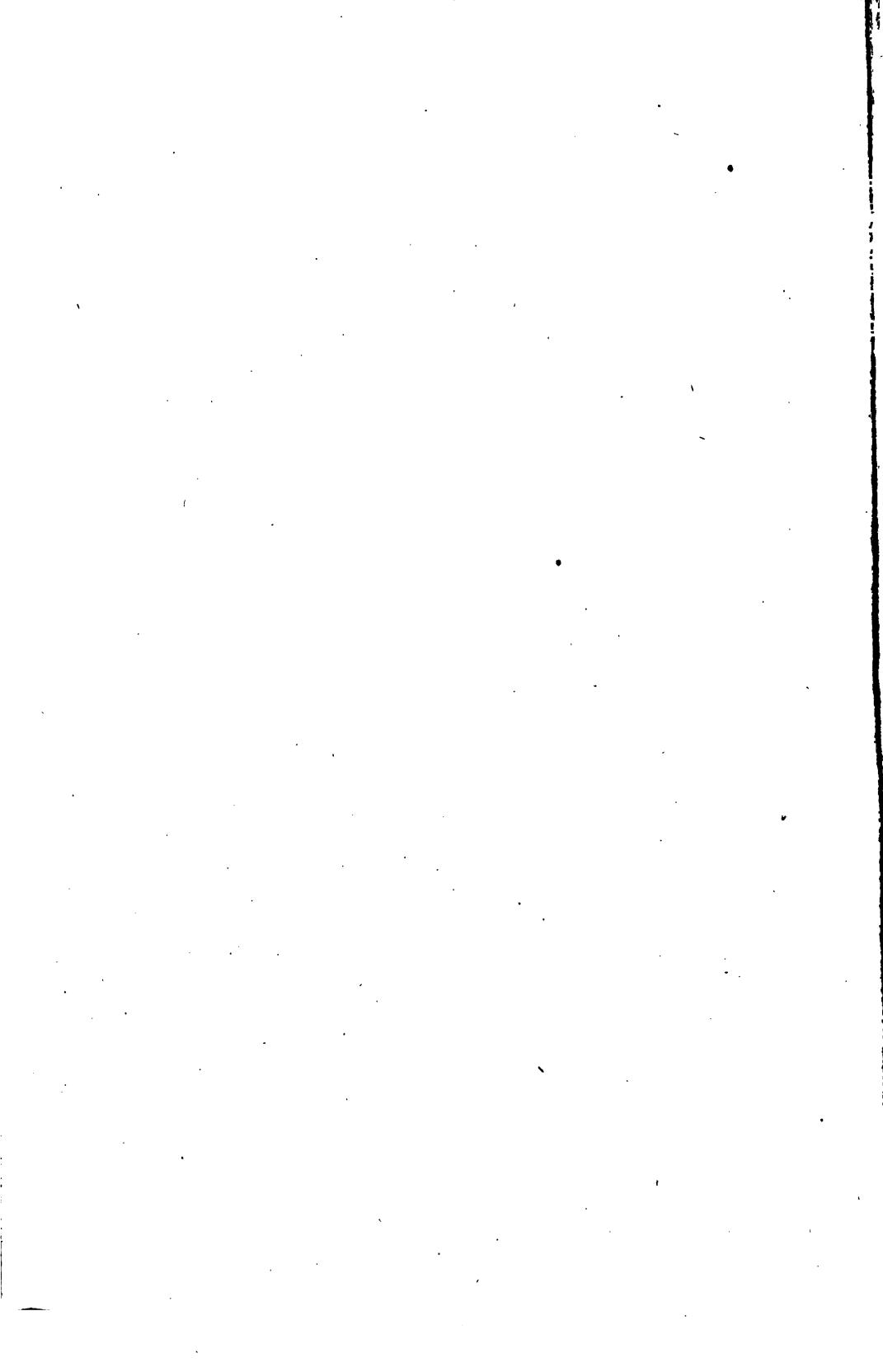
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H. W. WILEY, Chief of Bureau.

# COMMERCIAL TURPENTINES:

THEIR QUALITY AND METHODS  
FOR THEIR EXAMINATION.

BY

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AND

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WASHINGTON:  
GOVERNMENT PRINTING OFFICE.  
1911.

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## LETTER OF TRANSMITTAL.

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U. S. DEPARTMENT OF AGRICULTURE,  
BUREAU OF CHEMISTRY,  
*Washington, D. C., October 15, 1910.*

SIR: I beg to submit for your inspection and approval the results obtained in an investigation made in this Bureau by F. P. Veitch and M. G. Donk on the adulteration and grading of turpentines. The results show the extent to which turpentine is adulterated, and indicate the loss of values to the turpentine farmers, and the extent to which turpentine users are defrauded by adulteration. Methods for the analysis and testing of turpentine have been simplified and improved. Based largely on this investigation and the field work connected therewith, specifications for grades are suggested which it is believed will prove equitable to the producer and enable the purchaser to secure turpentine of the character desired. The data obtained are thus of special interest to the buyers and sellers of this product. I recommend that this report be published as Bulletin No. 135 of the Bureau of Chemistry

Respectfully,

H. W. WILEY,  
*Chief of Bureau.*

Hon. JAMES WILSON,  
*Secretary of Agriculture.*



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FIG. 1. Apparatus for the fractional distillation of turpentine.....	Page. 27
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# COMMERCIAL TURPENTINES.

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## INTRODUCTION.

### NATURE AND GENERAL PROPERTIES.

Commercial spirits of turpentine is a mixture of similar and closely related terpenes having the general composition expressed by the formula  $C_{10}H_{16}$ , with varying quantities of oxidized and hydrated derivatives of terpenes, usually present only in small amounts when first distilled from the gum.

These terpenes differ both in their physical and chemical properties, and consequently commercial turpentine may distill at from  $154^{\circ}$  to  $215^{\circ}$  C. ( $309^{\circ}$  to  $419^{\circ}$  F.). The fractions obtained on distillation have specific gravities ranging from 0.8580 to 0.9500 or more, with refractive indices of from 1.462 to 1.520, respectively.

The pinenes, several of which, differing slightly from one another in boiling point, specific gravity, and refractive index, have been recognized, are among the chief constituents of spirits of turpentine freshly distilled from the gum. Dipentene<sup>1</sup> has also been reported as occurring in American turpentine, but as dipentene distills alone at about  $178^{\circ}$  C., it is probably present in but small quantities in those turpentines which distill completely below this temperature. The above-mentioned terpenes boil between  $155^{\circ}$  C. ( $311^{\circ}$  F.) and  $178^{\circ}$  C. ( $352^{\circ}$  F.); their specific gravities lie between 0.845 and 0.866, and their refractive indices between 1.46 and 1.472. Other constituents which may be present, regarding the identity of which but little is known, have specific gravities of from 0.865 to 0.950 and refractive indices as high as 1.5200 at  $20^{\circ}$  C., and boil at from  $160^{\circ}$  C. to  $215^{\circ}$  C. ( $320^{\circ}$  to  $419^{\circ}$  F.). Turpentines which have been kept long, particularly those held in partly filled vessels, frequently have higher specific gravities, refractive indices, and boiling points than freshly distilled turpentine. In changing they become more oily and usually somewhat darker in color.

The quantity of each constituent present in commercial American turpentine is influenced not only by the physiological processes of the individual trees, but also, and probably more largely, by the

<sup>1</sup> Gildemeister, Die Aetherische Oele, 2d ed., 1910, p. 328; Allen, Commercial Organic Analysis, 3d ed., vol. 2, pt. 3, p. 263.

conditions under which the crude gum is gathered and the time which elapses before it is distilled, by the method of conducting the distillation, and finally by subsequent storage conditions. When the face of the tree over which the crude gum flows before it reaches the box is long, the weather hot, and the gum is kept long before it is distilled, there is more evaporation of the lighter constituents and at the same time the oxidation and polymerization of the gum are increased. It is customary to carry the distillation further on gum from old than from new boxes. The temperature in the final stages of the distillation rises higher, as a rule, and the product obtained near the close of the distillation is more frequently colored or burned. For these reasons turpentine from old boxes, as a rule, is heavier, distills less completely below 170° C., and has a higher refractive index than turpentine made from first-year gum or "virgin dip."

One of the chief causes of the observed variations in the constants of turpentine freshly distilled from the gum lies in the practice followed in the barreling of the product at the still. From time to time during the distillation the turpentine is transferred to barrels for shipment, and as a rule the turpentine first distilled is placed in one barrel, the middle portion in another, and the last off in a third barrel. In the earlier stages of the distillation the turpentine having the lowest specific gravity, refractive index, and distilling temperature passes over. As distillation proceeds the distilling temperature, gravity, and refractive index of the distilling turpentine steadily increase, though with occasional lapses and not necessarily proportionally. As a result the constants of the turpentine in the first barrel may agree with the lower limits, while that in the last barrel may approach the upper limits recognized for turpentine.

Because of these conditions, exposure in the woods, variations in distilling, differences in the product at successive stages of the distillation, and changes in composition after distillation, it is customary to recognize variations in the specific gravity of turpentine from 0.862 to 0.875, in refractive index from 1.4670 to 1.4750, both at 15.5° C., and in the initial boiling point of from 154° C. to 159° C. (309° to 318° F.), while the percentage which distills below 170° C. (338° F.) varies from about 85 to 99 per cent.

Old turpentine, even though it is kept in closed vessels, may undergo changes and have a specific gravity as high as 0.9460 and a refractive index of 1.480 or more, while the initial boiling point is raised and the percentage distilling below 170° C. (338° F.) is lowered.

It is desired in this bulletin to distinguish between old turpentine and turpentine to which mineral, coal tar, or other dissimilar oils have been added. While old, highly oxidized turpentines can not properly be classed as normal turpentines, they are, nevertheless,

mixtures of terpenes and related heavier oils and as such may possess the peculiar properties which are commercial assets of turpentine and which distinguish it sharply from unrelated oils which are often substituted wholly for it or employed in its sophistication. In the one case the product is a natural result of aging, while in the other it may properly be said to be adulterated, and such oils will be so distinguished in this publication.

#### SOURCES AND METHODS OF PRODUCTION.

Oil or spirits of turpentine, commonly known as "turpentine," or in the paint trade simply as "turps," is obtained chiefly from the longleaf pine (*Pinus palustris*), though a portion is also obtained from Cuban (*P. heterophylla*) and a little from loblolly pine (*P. taeda*). The Forest Service has found that loblolly, shortleaf, and Virginia pines yield naval stores equal in quantity to the longleaf yellow pines, and as the former occur in large quantities in the South their utilization would add largely to the turpentine resources of the country.

During the winter months before the sap begins to rise a pocket or "box" is cut in the tree near the ground, and at intervals during the succeeding spring and summer the bark and a portion of the sap wood are removed from the trunk above the box. This cutting greatly shortens the life of the tree and renders it more liable to destruction by wind, storm, and fire. The injury to standing timber caused by turpentining may be largely avoided by collecting in cups, the gum being directed into them by strips of zinc or heavily galvanized iron set in the face of the tree. This system, used exclusively in France, is now being extensively introduced in a modified form into this country and not only protects the tree but, as has been shown by the Forest Service, yields a higher grade of rosin and more turpentine.

In the spring the crude gum begins to flow from the "face," as that portion of the tree from which the bark and a part of the sapwood have been removed is called, and collects in the box or cup near the base, from which it is removed by dipping from time to time during the flowing season, which lasts from March to October or later. The thick, resinous mass, which usually contains water, chips, bark, pine needles, earth, and charred wood, is taken to the still, which is a large copper kettle with an outlet in the bottom by means of which it may be emptied and a cap connecting with a condensing coil. The kettle is bricked around in order that it may be safely heated by a fire underneath.

The crude gum is poured into the kettle until it is from one-third to one-half full and then slowly heated. A portion of the chips, bark, and pine needles is skimmed out after the contents of the kettle

become thoroughly liquid, and the cap is replaced in position and connected with the condenser while the heating of the kettle is slowly continued. Slow, cautious heating is made necessary by the presence of water in the gum, which causes frothing when the gum begins to boil, and if the heating is unduly hastened the gum will froth over, causing loss and delay. The presence of water in the gum lowers the boiling point of the mixture, so that both turpentine and water begin to distill at about 94° to 95° C. (201° to 203° F.); alone it distills at from 155° to 170° C. (311° to 338° F.).

When the danger of frothing over has passed, which means usually that most of the water has boiled off, a small stream of water is allowed to run into the still through the cap, as in this way the distillation is conducted at a much lower temperature than would otherwise be possible.

The condensed turpentine and water are collected in a separator, where the turpentine collects on top and may be drawn off from time to time. The separated spirits of turpentine is then run into tight white-oak barrels, which have been well driven and glued to prevent the turpentine soaking through the wood. The barrels are filled to within 1 gallon of their capacity, leaving room for expansion of the turpentine, which takes place with a rise of temperature and would cause leakage if the barrel were filled at the ordinary temperature. The barreled turpentine is then ready for the market.

The residue in the kettle from the turpentine distillation is rosin, which while still hot is run out of the opening in the bottom through wire screens and then strained through cotton cloth or batting to remove the remaining chips, straw, and dirt, after which it is barreled and cooled.

When distillation is properly conducted with water all of the turpentine distills below 150°-160° C. (302°-320° F.), and there is little apparent breaking up of rosin or discoloration of the turpentine. If the temperature is allowed to rise above 160°-170° C. (320°-338° F.) toward the end or the distillation is prolonged, or if water is not used in distillation, both the turpentine and the rosin are darker colored.

The color of turpentine is due primarily to overheating the gum and possibly partly to the chips, pine straw, etc., left in the kettle during the distillation. Wood begins to color decidedly above 150° C. (302° F.), so that it is almost impossible to make a colorless rosin if there are any chips or bark in the still. Prolonged heating of the rosin at 130° C. will lower the color several grades. Hence, to make turpentine and rosin having but little color the gum should be free of wood, bark, and pine straw, the contents of the still should never rise above 160° C. at any point, and the distillation should be conducted as rapidly as is practicable.

In the past but few stills were equipped with thermometers to indicate the temperature of the gum in the still, but it is probable that in properly conducted distillation the temperature rarely rose above 170° C., and for virgin gum was considerably lower than this. Thermometers are coming into more general use in turpentine distilling, and the danger from overheating is being correspondingly reduced.

#### DISTRIBUTION, QUANTITY PRODUCED, AND VALUE.

The turpentine-producing area in this country is practically confined to the coastal plains region of the Southern States. In the earlier days the industry was best developed in North Carolina, but owing to destructive methods of turpentine orcharding, in conjunction with lumbering, fires, etc., the industry has gradually worked southward and westward, until at present Florida produces the most turpentine, followed by Georgia, Alabama, Mississippi, Louisiana, North Carolina, South Carolina, and Texas, in the order named.

The statistics of the production for the past 40 years taken from the census reports are given in the following table:

*Turpentine production in the United States for the past 40 years.*

Year.	Gallons.	Value.
1870.....	6,004,887	\$2,194,498
1880.....	17,566,250	1,542,120
1890.....	17,316,200	5,459,115
1900.....	38,488,170	14,980,235
1905.....	30,687,051	15,170,499
1908.....	36,589,000	14,112,400

#### GRADING, MARKETING, AND USES.

It is the trade practice to grade turpentine according to its color, and the various grades are known as "water white," "standard," "off one shade," "off two shades," and "off three shades." The latter is not merchantable. Under the trade regulations the deduction in price on turpentine off one shade is 2.5 cents per gallon and off two shades 4 cents per gallon. Of late years, however, it has become customary to mix the colored turpentine with water-white or standard turpentine, adding a small quantity to each barrel which is not full. The factor is paid 50 cents for each barrel of the colored turpentine that he uses in this way. By this practice the producer receives more for his colored turpentine than he otherwise would under the trade regulations, and but one grade of turpentine is generally known to the buyer beyond the primary markets. There are, however, as has been indicated in the discussion on general properties and methods of production, decided differences in the

quality of various lots of turpentine. Recognizing this fact many users of large quantities buy on definite and rather strict specifications, and it would probably be of advantage to the trade if several grades for turpentine were more generally recognized.

About half of the turpentine produced in this country each year is exported, chiefly to Europe, while by far the larger part of the remainder is consumed in the varnish and paint industries, and small quantities are used in medicine and as a solvent for gums, rubber, fat, and waxes, in this country. No statistics are obtainable showing the quantities consumed for the latter purposes, nor in thinning paints and varnishes at the time they are actually used. The distribution of turpentine produced in 1905<sup>1</sup> was as follows:

	Gallons.
Produced.....	30,687,051
Exported.....	15,894,813
Used in manufacture of paints and varnishes.....	7,160,774
Used in medicine, and as a solvent in other industries and for paint and varnish thinning, etc. (by difference).....	7,631,464

#### COLLECTION AND CLASSIFICATION OF SAMPLES.

The turpentine found on the American market is quite frequently adulterated with cheaper and inferior oils, those most commonly employed being the petroleum oils having specific gravities corresponding closely to that of turpentine, and distilling at about the same temperature. Other adulterants are certain coal-tar oils, rosin spirits, and wood turpentine, which closely resemble turpentine in specific gravity, distilling temperature, and some other properties.

Work on turpentine found on the American market was undertaken under the Food and Drugs Act of June 30, 1906, for the primary purpose of determining the nature and extent of adulteration in turpentine sold for medicinal purposes. Furthermore, it is desirable to know at what stage in the marketing of this article it is generally adulterated. The naval stores industry is vitally interested in the matter, as the value of the turpentine output is materially affected by the practice of adulteration. It is quite common for druggists, both wholesale and retail, to buy turpentine from oil and paint dealers or from anyone handling it in large quantities, and, therefore, samples have been secured from oil and paint dealers, as well as from druggists, their stock being in many instances representative of the turpentine sold at the drug stores in the same community. In fact rarely is any distinction made between turpentine for drug or technical purposes.

Though it is known that spirits of turpentine is very frequently adulterated, no systematic investigation of the subject in this

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<sup>1</sup>Later statistics on the quantity used in manufacture of paints and varnishes are not available.

country is on record. For this reason, the results obtained on samples collected from all parts of the country are recorded for the information of the public, to show the extent of the practice of adulteration, and to furnish more complete data for the grading of American turpentine and the preparation of specifications therefor which shall be fair to the producer and also insure that the consumer secures the article for which he pays.

The samples have been classified into those secured within and without the turpentine-producing States, and each of these classes has been subdivided into three groups. First, samples taken from the producer, or in the yards of primary buyers, but which the primary buyers stated, and the evidence indicated, were in the original containers in exactly the condition in which they were received from the producer. Second, samples taken from the tanks or other containers of primary buyers after they had accepted the goods from the producers. Third, samples taken from the stock of wholesale and retail druggists, paint and hardware merchants, and other dealers. These classifications are made to show as clearly as possible at what point in the turpentine industry adulteration is most practiced.

#### **METHODS OF ANALYSIS EMPLOYED AND THEIR DISCUSSION.**

##### **GENERAL EXAMINATION.**

Owing to the nature of turpentine, there are quite wide variations in its chemical and physical constants, and as these natural variations do not appear within limits to affect its suitability for technical and medicinal purposes, rigid control of analytical procedure usually is not insisted upon in the examination of the article, and, indeed, great refinement of procedure is scarcely warranted. Thus specific gravity and refractive index need not be expressed beyond the third decimal, flash point and distilling temperature should be stated in whole degrees, while the residues from polymerization and the results of distillation should be given in whole numbers as percentage. For these reasons, and in order that the results might be comparable with other published data, the usual procedure in the examination of turpentine has been followed in the main in analyzing the samples reported in this bulletin. It became apparent, however, as the work progressed that certain precautions and modifications in making such examinations were desirable in order that concordant results might be obtained by different analysts, and conditions affecting the results on adulterated and certain oxydized samples be better controlled. The details of the modified methods are given, beginning on page 26.

The methods as applied to the samples analyzed in this investigation are as follows:

*Specific gravity* was determined with the Weetphal balance at 20° C.

*Refractive index* was determined at 20° C. with the Zeiss refractometer.

*Color* was determined by means of the Lovibond colorimeter, and is, for simplicity of standardization, expressed in terms of the depth in millimeters of the liquid which gives a color equal to the Lovibond tintometer glass No. 1 yellow.

*Distillation*.—One hundred and fifty cubic centimeters of turpentine were put in a 200 cc flask having a neck 20 mm in diameter and the side tube 10 cm from the bulb. The bulb of an accurate thermometer was placed opposite the side tube, the flask set in a glycerin or oil bath, connected with condenser, heated, and distillation conducted at the rate of two drops per second.

The results obtained on the commercial samples have not been corrected for the emergent stem of the thermometer, and for this reason the recorded readings are on the average, by actual comparison, about 3.5° lower, and as a maximum on the higher boiling fractions, about 6° lower, than the actual temperatures. Neither has correction been made for variation in barometric pressure. The most serious consequence of not making these corrections is found in determining the exact initial distilling point and the percentage of turpentine distilling at successive temperatures. As a matter of fact, these corrections would have but little effect in the case of a normal turpentine on the total percentage distilling below the upper limit for turpentine, which may very properly, as the work indicates, be put at 170°, when the reading is taken with the scale of the thermometer entirely within the neck of the distilling flask.

#### EFFECT OF EMERGENT THERMOMETER.

The differences in temperature readings, due to emergent stem, and the effect of these differences on the quantity of turpentine distilled at various temperatures as determined by actual comparisons, are shown in the following table. In all cases the bulbs of the thermometer were opposite the side neck of the flask.

*Comparison of differences of temperature and of volumes distilled, due to emergent stem of thermometer.*

[200 cc of turpentine distilled.]

Turpentine No. 1.				Turpentine No. 2.			
Reading of thermometer—		Difference.	Volume distilled.	Reading of thermometer—		Difference.	Volume distilled.
Immersed. <sup>1</sup>	Emergent.			Immersed. <sup>1</sup>	Emergent.		
°C.	°C.	°C.	cc.	°C.	°C.	°C.	cc.
157.0	153.5	3.5	.....	156.0	151.0	5.0	.....
157.4	154.0	3.4	7	158.0	154.0	4.0	.....
158.2	155.0	3.2	26	159.2	155.0	4.2	12
160.0	156.5	3.5	108	160.2	156.5	3.7	28
164.0	160.0	4.0	166	165.0	162.0	3.0	83
165.0	162.0	3.0	173	167.0	163.0	4.0	99
170.0	166.0	4.0	183	169.4	165.0	4.5	131
172.0	167.0	5.0	184	170.0	166.0	4.0	136
.....	.....	.....	.....	175.0	170.0	5.0	151
.....	.....	.....	.....	177.0	172.0	5.0	155
.....	.....	.....	.....	180.0	174.0	6.0	159
.....	.....	.....	.....	185.0	178.0	7.0	163
.....	.....	.....	.....	190.0	183.0	7.0	166
.....	.....	.....	.....	195.0	187.0	8.0	168
.....	.....	.....	.....	200.0	192.0	8.0	170

<sup>1</sup> Thermometer immersed in vapor only.

The table shows that No. 1 turpentine begins to distill at 157° C. (315° F.) by the immersed thermometer and at 153.5° C. (308° F.) by the emergent thermometer. At 160° C. (320° F.) by the immersed, 108 cc had distilled, and by the emergent thermometer 166 cc. At 165° C. (329° F.) by the immersed thermometer 173 cc had distilled, while about 180 cc had passed over when this temperature was reached by the emergent thermometer. It will be noted also that as the temperature rises, the difference between the readings of the two thermometers first decreases and then increases; the difference in reading being least where approximately the largest volume of distillate is yielded. These facts are brought out still more strikingly in the distillation of turpentine No. 2. The lag of the emergent stem thermometer is also shown in the comparison of the initial distilling temperature. The calculated correction for the emergent stem thermometer at 160° is 2°, while the actual correction as determined by experiment was never less than 3°. These figures show how necessary it is, if comparative data as to the progress of a distillation are desired to have the stem of the thermometer entirely immersed in the vapor, and they also indicate that corrections made under ordinary laboratory conditions for emergent stem and lag are likely to be inadequate.

#### EFFECT OF VARIATIONS IN ATMOSPHERIC PRESSURE.

Variations in atmospheric pressure may affect the volume of the distillate at any given temperature even more than errors introduced by reading temperatures on emergent thermometers. The effect of variations in pressure on the distillation of two turpentines is shown in the table on page 16.

*Variations in amounts of turpentine distilled caused by different barometric pressures.*

[200 cc of turpentine distilled.]

Turpentine No. 1.				Turpentine No. 2.			
Temper- ature.	Amounts distilled at different baro- metric pressures.		Differ- ence.	Temper- ature.	Amounts distilled at different baro- metric pressures.		Differ- ence.
	740 mm.	780 mm.			740 mm.	780 mm.	
°C.	cc.	cc.	cc.	°C.	cc.	cc.	cc.
157.4	7	.....	.....	159.2	12	.....	.....
159.0	53	10	93	160.0	26	12	14
160.0	108	10	98	165.0	99	60	33
165.0	173	163	10	170.6	136	120	13
170.0	183	180	3	175.0	151	143	8
	.....	.....	.....	180.0	159	156	6

These results show that in the early stages of the distillation the quantities distilled at the two pressures differed widely; more widely on the turpentine distilling at the lower temperatures and within the narrower limits. As distillation approached completion these differences decreased, becoming negligible when 90 per cent of No. 1 had distilled, but remaining large when 80 per cent of No. 2 had passed over.

The distillation of normal fresh turpentine, of which more than 80 per cent passes over below 165° C. (329° F.), is greatly affected by variations in pressure; and it is quite clear that in obtaining the initial distilling temperature and other distillation data at temperatures below 165° C., the pressure should always be the same. As distillation proceeds and the temperature rises the effect of variations in pressure on the total amount of turpentine distilled becomes less, so that ordinarily (that is, on a turpentine 90 per cent or more of which distills below 170° C.) when the distilling temperature rises to 170° C. the total quantity distilled at 740 mm or at 780 mm is almost the same, and for all practical purposes the difference may be disregarded. When the percentage distilling above 170° C. is large, however, as in the case of No. 2, the difference due to the variation in pressure is large and not to be ignored. If it is desired, therefore, to determine the percentage distilled below 170° C. to determine accurately the initial boiling point, or to trace the course of the distillation throughout its entire range, the barometric pressure should always be corrected to 760 mm.

#### DETECTION OF ADULTERANTS.

The majority of the samples analyzed were examined only for mineral oils, as these are the principal adulterants employed. Methods for the detection of certain other adulterants were studied, however, and the results obtained will be discussed. Wood turpentine was not detected in any of the samples examined.

## SIGNIFICANCE OF ODOR, COLOR REACTIONS, AND BEAD.

Straight wood turpentines are readily distinguished from gum turpentines by their odor, or when they have been very carefully refined, by the odor of the first fraction, or of the residue from fractional distillation. One or both of these portions have the peculiar "sawmill smell," and the residue has a camphoric and somewhat nauseating odor characteristic of wood turpentine, which is quite different from the mild, sweet fragrance of gum turpentine.

Destructively distilled wood turpentine and also rosin spirits are more readily distinguished from gum spirits by their odor than is wood turpentine prepared by steam distillation, and they also give distinguishing color reactions when mixed with sulphurous acid as suggested by Hertzfeld or with hydrochloric acid (the presence of tin or zinc is not necessary) as suggested by Grimaldi. When steam-distilled wood turpentine has been carefully refined so that 90 to 95 per cent of it distills at from 155° to 170° C. (311° to 338° F.), both of these color tests are practically useless.

Turpentine adulterated with more than 10 to 20 per cent of coal-tar oils, or of gasoline or kerosene which have not been deodorized, may usually be readily detected by the characteristic odor of the mixture. The odor of rosin spirits, while quite distinctive, is difficult to detect in mixtures with turpentine. The presence of petroleum oils is also indicated by bubbles or "beads" persisting for a few moments on the surface of the turpentine shaken in a partly filled bottle.

The presence of more than about 10 per cent of kerosene or similar mineral oils is readily detected by the spot which a few drops of the sample placed on white paper leaves on drying. Gasoline and other light mineral oils do not leave this spot.

## MINERAL OIL.

It is possible so to adulterate turpentine that neither the specific gravity, refractive index, or flash point is materially altered. Such careful adulteration is, of course, very rare, but as adulterants can not invariably be detected by making these determinations, and further, as mineral oil is the most probable adulterant, it is more rational to at once polymerize the suspected sample, note the volume, color, and consistency of the unpolymerized residue, and determine its refractive index.

The detection of mineral oils in turpentine is relatively quite simple, and is accomplished by mixing the turpentine with a certain proportion of sulphuric acid of a given strength, in which the turpentine is destroyed and mixes with the acid while most of the mineral oil remains unaffected and separates in a layer on top of the acid. The polymerization or sulphonation of turpentine, as this operation is

called, as improved by Mr. Donk and conducted in the Bureau of Chemistry, is as follows:

Place approximately 25 cc of exactly 38 times normal sulphuric acid in a flask holding approximately 50 cc and having a graduated neck. The ordinary Babcock milk-test bottle serves admirably, and being a stock article is readily obtainable. Cool the flask in ice and run in without mixing 5 cc of the turpentine to be tested. If the turpentine is pure and recently distilled, the charred surface will have a green fluorescence when held to the light and the underlying sulphuric acid will be clear and slightly colored. As little as 5 per cent of mineral oil prevents the green fluorescence and gives the sulphuric acid a muddy appearance. Gum and wood turpentine containing considerable quantities of heavy terpenes also lack fluorescence and are muddy in appearance. Mix the turpentine with the acid by cautious shaking, keeping the temperature below 65° C. (149° F.) by frequent immersion in ice. When the temperature no longer increases on shaking (determine by the touch), shake vigorously to insure perfect contact between the acid and the turpentine, place the flask in water at about 40° C. (104° F.), and heat slowly to 65°, shaking vigorously and frequently. When this temperature is attained, give a final vigorous shaking; cool, bring the unpolymerized layer into the neck by running in ordinary sulphuric acid, centrifuge or let stand for two hours, read the volume of unpolymerized clear top layer and immediately determine its refractive index, withdrawing a portion from the top of the clear layer only for this purpose. The character and quantity of this layer or residue will show whether or not the turpentine is adulterated with mineral oil. If the turpentine is pure, the residue will seldom exceed 1 per cent; it will be very viscous, and will have a refractive index at 20° C. of 1.5000 to 1.5200. If the sample contains mineral oil, however, the residue will be limpid or oily and colorless and will have a refractive index at 20° C. between 1.4200 and 1.4950.

It is characteristic of mineral oils that the refractive index of the unpolymerized oil is lower than that of the original sample, while that of the residue from turpentine is higher. The results are not quantitative, as when 5 per cent of mineral oil is present approximately 80 per cent of it is taken up by the sulphuric acid, when 10 per cent of mineral oil is present approximately 60 per cent, and when 20 per cent is present approximately 50 per cent of mineral oil is destroyed in polymerization. When a straight mineral oil is treated with the 38 normal sulphuric acid, about 25 per cent of it is acted upon. For kerosene these figures are much lower. In this report residues greater than 1 per cent are considered to be mineral oil and no correction is made for the amount destroyed in polymerization. These results are therefore low, giving the benefit of the known action of the acid on the mineral oil entirely to the seller.

In a number of instances when the volume of the residue after polymerization did not indicate the presence of mineral oil but its refractive index or limpidity did, the turpentine was distilled, and the portion which did not distill below 170° C., and several of the fractions from the distillation were subjected to the sulphuric-acid test. As a rule, the portion which did not distill below 170° C. left more residue after polymerization than the original turpentine did and it possessed more decidedly the characteristics of mineral oil, i. e., low

refractive index and limpidity. The undistilled portion of pure turpentine when treated in the same way either foamed out of the flask or was instantly reduced to a charred mass and left no measurable residue.

#### ROGIN SPIRITS.

If rosin spirits is present in notable quantities, it may be detected by the amount of residue on polymerization in conjunction with lowered initial boiling point and the deep coloration produced by mixing portions of the original turpentine or the first fraction with sulphurous acid and with hydrochloric acid.

If the initial distilling temperature of the turpentine is less than 154° C. and the original sample gives a reddish color with sulphurous acid and a greenish red with hydrochloric acid, and the residue from polymerization is less than 5 per cent,<sup>1</sup> it is probable that the sample is adulterated with rosin spirits. An odor of rosin spirits in the sample, or in any fraction, would be confirmatory, and all tests should be repeated on the first fraction, obtained with a fractionating column. At best the detection of small amounts of rosin spirits is very difficult, and the procedure outlined may often fail to detect its presence.

#### COAL-TAR OILS.

The presence of marked quantities of coal-tar oils, benzol, toluol, and xylol is detected by the odor, the lower initial distilling temperature, i. e., between 80° and 155° C. (176° and 311° F.), and by further examination of the first fractions which distill below 155° C. (311° F.). The rise of temperature on shaking during polymerization is also indicative of the nature of the sample. Coal-tar and petroleum oils do not raise the temperature more than 35° or 40° C. (95° or 104° F.), when mixed with sulphuric acid. When the temperature of turpentine mixed with 38 normal sulphuric acid does not rise above 50° or 60° C. (122° or 140° F.), it is evident that mineral oil, coal-tar oil, or some other oil than turpentine is present in large quantities. The older the sample of turpentine, the greater, or at least the more rapid, the rise of the temperature of the mixture. When the initial distilling temperature of the sample is less than 154° C. (309° F.) and there is less than 1 per cent of residue on polymerization, the presence of coal-tar oils is to be suspected. The sample should be carefully fractionated with a column, the initial distilling temperature noted, and that portion distilling below 157° C. (315° F.) carefully examined. A refractive index at 20° C. greater than 1.475 is strongly indicative of coal-tar oils and the test should be repeated, distilling a sufficient quantity of the sample to obtain at least 50 cc fractionating below 157° C. (314° F.), which should be

<sup>1</sup> Rosin spirits leaves a small residue on polymerization.

carefully fractionated, the initial distilling temperature noted, and the refractive index, behavior with sulphuric acid (color, heat developed, refractive index of residue, if any), and odor closely observed. An initial distilling temperature of less than 154° C. (309° F.) with a refractive index of the first fraction of more than 1.475, together with a reddish color and but little development of heat on mixing with the sulphuric acid, and the characteristic odor of the oils, is proof of the presence of coal-tar oils.

When the proportion of coal-tar oils is small, this examination may not always give positive evidence of their presence. As little as 5 per cent of xylol, however, may be identified by the following procedure:

To 150 cc of 4 to 1 sulphuric acid, in a separatory funnel, add 100 cc of the suspected turpentine, being careful to prevent a rise in temperature above 40° C. (104° F.). Mix thoroughly, keeping the temperature below 40° C., draw off the polymerized portion, transfer the unpolymerized portion to a small distilling flask, and distill slowly, changing the receiver when the distillate ceases to be clear, colorless, and limpid. The temperature at this time should not exceed 180° C. (356° F.). A turpentine containing 5 per cent of xylol, when treated in this way, gave about 3 cc distilling below 180° C. (356° F.). The refractive index was 1.475, the odor that of xylol, and on adding nitric acid a strong odor of nitro-xylol was obtained. This treatment of turpentine with dilute sulphuric acid converts it into a heavy oil (with a refractive index of 1.5+), and not more than a few cubic centimeters will distill from the mixture below 300° C. (572° F.). The method has not so far been sufficiently developed to be of quantitative value, so that the proportion of coal-tar oils present can only be approximated within wide limits.

Nonvolatile oils are, of course, readily detected by distillation, and their nature, so far as it may be determined, is learned by the usual methods.

#### DISCUSSION OF DATA ON UNADULTERATED SAMPLES.

It will be observed in studying the detailed analytical data that a few samples, which undoubtedly do not contain added adulterants, show some inconsistencies among the figures for specific gravity, refractive index, and behavior on distillation. While, as has been mentioned, this fact is partially accounted for by the use of emergent stem thermometers and the variation in barometric pressure, it is certainly not always due entirely to these causes, but also partly to the fact that, in examining so large a number of samples, it was impracticable to make all the determinations on each sample at one time, and changes occurred in these few turpentines in the time that elapsed between the beginning and the ending of their analysis. For some of

the samples two sets of results are given. They show that while some have undergone no alteration even after 18 months others have continued to change. All of the figures on the latter samples are of interest as indicative of the changes which may take place in turpentine during aging. The results on the turpentine which had evidently changed greatly were not considered in forming a judgment regarding the individual constants of turpentine.

The following summary of the analytical data on unadulterated turpentine, based on the detailed figures given in Tables I and II, shows the extremes obtained:

*Summary of analytical data on unadulterated turpentine.*

Determinations.	Minimum.	Maximum.
Initial distillation temperature (° C.).	154.5	159.0
Distillation below 160° C. (per cent).	42.0	94.0
Distillation below 165° C. (per cent).	68.8	98.6
Distillation below 170° C. (per cent).	77.3	99.1
Specific gravity.	.8617	.8989
Refractive index.	1.4684	1.4818
Color (mm.).	2.0	800.0
Polymerization residue (per cent).	.2	1.0
Refractive index of polymerization residue.	1.500	1.5150

In setting these limits the results on several samples have been ignored, because there was doubt as to the purity of the sample, or as to the exactness of the results.

The summarized data show that turpentine which are unadulterated, in the sense that they have had nothing added to or taken from them, give widely different results when analyzed under the usual conditions. Thus unadulterated turpentine may begin to distill, under the conditions obtaining in this work, at from 154.5° to 159° C. The amount distilled below 160° C. varies from 42 to 94 per cent, below 165° from 69 to 99 per cent, and below 170° from 77 to 99 per cent. The specific gravity ranges from 0.8617 to 0.8989 at 20° C. and the refractive index from 1.4684 to 1.4818. The residue on polymerization with 38 normal sulphuric acid ranges from 0.2 to 1.0 per cent, and the refractive index of this residue at 20° C. may be as high as 1.5200 but should never be below 1.500. These differences are due to natural variations in the freshly distilled turpentine, to aging, and to variations in conditions of analysis.

It should be borne in mind that many samples of old turpentine are undoubtedly represented in this lot, as well as turpentine stored in wood and in metal, and in clean containers as well as in tanks which still held the residues of previous purchases. In other words, the samples fairly represent those found on the wholesale and retail market at any time. Certain of these samples can not be properly termed turpentine because they do not possess the constants of turpentine.

It has been pointed out that in aging, turpentine undergoes change; it becomes heavier, thicker, and deeper colored and the odor is different. With age specific gravity and refractive index may become higher, the quantity distilling below 170° C. less, the color deeper, and polymerization with sulphuric acid more rapid, and fully as complete. The increase in specific gravity or refractive index which occurs with age is well shown by the results on samples Nos. 6121, 6127, 6135, 6137, and 6238, on which two sets of results, the higher one obtained from 12 to 18 months later than the lower, are given. These samples had visibly changed in consistency and color, and the odor had also changed in the time elapsing between the two sets of determinations. Samples which showed no visible darkening or thickening had practically the same specific gravity and refractive index after 18 months as at first. As has been shown, partly because of this tendency to change with age and also because of the natural variations in the product, it is customary to specify certain limits within which the results of an analysis of turpentine should fall. The fixing of these limits for different grades is discussed under specifications for turpentine.

#### EXTENT OF ADULTERATION WITH MINERAL OIL.

The data showing the extent and distribution of the adulteration of turpentine, which is practically confined to the addition of mineral oils, may be summarized as follows, the detailed figures being given in Tables I and II, beginning on page 32. Only those samples showing 1 per cent or more of mineral oil are counted as adulterated in this summary:

*Number and distribution of adulterated samples.*

Source of samples.	Number.	Number adulterated.	Per cent adulterated.	Per cent of adulterant present.
<b>Samples taken in turpentine belt:</b>				
Producers' goods.....	71	3	4.2	2.9
Primary buyers' goods <sup>1</sup> .....	44	6	13.6	2.2
Dealers' goods.....	99	18	18.0	6.4
<b>Totals and averages.....</b>	<b>214</b>	<b>27</b>	<b>12.6</b>	<b>5.0</b>
<b>Samples taken outside of turpentine belt:</b>				
Primary buyers' goods <sup>1</sup> .....	7	0		
Dealers' goods.....	205	50	24.4	10.6
<b>Totals and averages.....</b>	<b>212</b>	<b>50</b>	<b>24.0</b>	<b>10.6</b>
<b>General totals and averages.....</b>	<b>426</b>	<b>77</b>	<b>18.0</b>	<b>6.1</b>

<sup>1</sup> Buyers purchasing through factors.

The figures indicate that only a few of the samples of producers' goods are adulterated with mineral oils, while the turpentine of the primary buyers and dealers in the turpentine belt is adulterated extensively, showing 13.6 and 18 per cent of adulterated samples,

respectively. The dealers' samples contain a much higher percentage of the adulterant than either of the other two classes, exceeding the producers' goods by 3.5 and the primary buyers' goods by 4.2 per cent. It will be observed also that adulterations occur more frequently and are present in larger quantities outside the turpentine-producing belt than within it. Eighteen per cent of all the samples collected were adulterated, and the average percentage of adulterant in these was 6.1 per cent, ranging from 1 to 71 per cent in the individual samples. The average amount of mineral oil in a barrel of the adulterated samples was approximately 3 gallons, which, basing the calculation simply on the cost of the two materials, made these adulterated turpentines worth fully \$1.50 less per barrel than unadulterated stock. These facts assume graver significance when it is remembered that turpentine is an article so well known and so generally used that it is carried in stock and sold by merchants generally, particularly by country merchants, and when bought from any source is liable to be employed indiscriminately, either medicinally or technically.

These samples having been carefully taken from all sections of the country, this statement regarding the percentage of adulterated turpentine may be considered as fairly representative of the general conditions in the turpentine trade. The consumption of turpentine in the United States during 1908 was approximately 15,000,000 gallons. If 18 per cent of this was adulterated, then approximately 3,000,000 gallons of adulterated turpentine, containing an average of at least 6 gallons per hundred of mineral oil, costing about one-fifth as much as the turpentine, was sold at turpentine prices. The small percentage of mineral oil found in some samples may have been occasioned by mixing a barrel of heavily adulterated turpentine with a large quantity of a pure product, or possibly by putting turpentine into improperly prepared petroleum barrels or into tank cars previously used for mineral oils.

#### EXAMINATION OF STANDARD GRADE SAMPLES.

The proper grading of turpentine is a matter of no small monetary importance to the producer and primary buyer, and the boards of trade of the turpentine markets have adopted rules and standards for the purpose of correct and consistent grading. These standards are selected by the naval stores inspectors and passed upon by the naval stores committee of the boards of trade. The generally accepted final authority is the New York naval stores committee, whose inspector, as a matter of fact, selects practically all standard samples of turpentine and rosin used by naval stores inspectors of this country.

For the purpose of learning how closely the standard samples on which turpentine is bought in the primary markets agree among themselves, sets of standards were secured from the Savannah, Ga., and

Jacksonville, Fla., boards of trade and also from the inspectors of the New York naval stores committee.

The color of these samples was read against the standard yellow glass on the Lovibond tintometer, marked "1." The turpentine was placed in a graduated 200-mm tube and the depth of the column of turpentine required to equal the color of the glass was carefully noted, using the Schreiner type of colorimeter. The longer the column of turpentine required to match the glass, the less color there is in the turpentine. The results are expressed in millimeters, which show the length of column or depth of turpentine required to equal the standard yellow glass No. 1. The following results were obtained on these standard samples:

*Comparison of standard turpentine samples from different sources.*

Sample marked—	Savannah sample.	Jacksonville samples.		New York sample.
		No. 1.	No. 2.	
Water white.....	mm.	mm.	mm.	mm.
Standard.....	1180	192	180	150
Off 1 shade.....	36	28	33	25
Off 2 shades.....	16	22	20	12
Off 3 shades.....	4	4	12	6
Off 4 shades.....				4

<sup>1</sup> This sample probably should have been marked "water white."

These results show that the standard samples obtained from different sources are far from uniform and indicate different practices on the chief primary markets. It is the usual custom, however, as previously stated, to mix small quantities of off-shade turpentine with the standard or water-white products and thus the off-shade output is used and sold without getting beyond the primary markets. The variations noted in the standards have, therefore, but little significance in the retail trade where, in general, but one grade of turpentine is recognized, though, as will be seen by consulting the tables, there is a great variation in the color of commercial turpentine, which point as an indication of quality should undoubtedly receive more careful consideration from the consumer than it does.

#### PROPOSED SPECIFICATIONS FOR TURPENTINE.

Buyers of large quantities of turpentine have long specified certain tests with which the turpentine they purchased must comply. In the main, most of these are reasonable and will be met by carefully made turpentine when freshly distilled. The constants and standards thus set differ slightly in different specifications, and some of them are unnecessarily severe, causing the rejection of much good turpentine. It seems well, therefore, to suggest specifications for

turpentine, which, while fully protecting the consumer, recognize the natural variations occurring even in the freshly distilled turpentine.

This investigation, however, shows clearly that by no means all of the unadulterated turpentine sold can be regarded of the same quality, nor does it comply with the accepted standards. This fact is fully accounted for by natural differences in the crude turpentine, negligence on the part of the turpentine producer, irregularities in distilling, and differences in age and methods of storing. A turpentine with a specific gravity of 0.875, only 50 per cent of which distills below 160° C. and 20 mm of which equals in color the No. 1 Lovibond glass, is certainly not of the same grade as one having a specific gravity of 0.865, 90 per cent distilling below 160° C. and 150 mm of which is needed to equal the No. 1 glass in color. Nevertheless the former is turpentine, though its value is somewhat less than the latter. It appears but just and reasonable that several well-defined grades of turpentine should be recognized and standard specifications established for them. Such standards are accordingly suggested for the consideration and criticism of producers and consumers of turpentine.

Standard or No. 1 turpentine should have a specific gravity at 20° C. of from 0.862 to 0.870; a refractive index at 20° C. of from 1.468 to 1.476; 95 per cent should distill below 170° C., and a layer of not less than 200 mm should be required to equal in color the Lovibond yellow glass No. 1. On polymerization with 38 normal sulphuric acid the residue should not exceed 1 per cent, should be reddish in color and viscous, and its refractive index at 20° C. should be from 1.500 to 1.520. An unadulterated turpentine which does not agree with these requirements may properly be regarded as not of standard or No. 1 quality.

Second quality or No. 2 turpentine should have a specific gravity at 20° C. of from 0.862 to 0.875; a refractive index at 20° C. of from 1.468 to 1.480; 90 per cent should distill below 170° C. and a depth of not less than 100 mm should be required to equal the Lovibond yellow glass No. 1. The polymerization residue must not exceed 1 per cent and must have a refractive index of not less than 1.50.

Third quality or No. 3 turpentine should have a specific gravity at 20° C. of from 0.865 to 0.880; a refractive index at 20° C. of from 1.468 to 1.485; 60 per cent should distill below 170° C. and a depth of not less than 50 mm should be required to equal the Lovibond yellow glass No. 1. The polymerization residue must not exceed 1 per cent and must have a refractive index of not less than 1.500.

These specifications are applicable to both gum spirits turpentine and to "wood turpentine," and are in general harmony with trade practice. For high-grade varnishes and for medicinal purposes a colorless, light turpentine which evaporates rapidly and will hasten drying, leaving the varnish firm and not "tacky," is desired. For

lower-grade varnishes and good quality paints a somewhat colored and slower evaporating product may be successfully employed. In the case of low-grade paints, especially those where the drying is hastened by vigorous means, or in all cases when slow drying is desired, the heavier, slower evaporating turpentine, or even what is known as "pine oil," may prove most suitable.

#### METHODS FOR THE EXAMINATION OF TURPENTINE FOR PURITY AND QUALITY.

The work done in connection with this investigation has developed a number of improvements in the methods for examining turpentine which increase the accuracy of the results and insure greater concordance in the work of different chemists. The methods to be given have been in use in this laboratory for from one to three years and have been found satisfactory.

All tests of a turpentine should be promptly made, usually within the same week, because some samples change rapidly in consistency, color, gravity, behavior on distillation, and refractive index, and if the several determinations are made at long intervals those last made may not represent the turpentine as first received. Further, all of the results on a sample may not be strictly comparable and may lead to errors in judging the turpentine.

#### COLOR.

Determine color in a graduated 200 mm tube in a colorimeter reading against the Lovibond yellow glass No. 1. If a depth of 200 mm or more is required to match the glass the color is No. 1; if a depth of from 100 to 200 mm is required the color is No. 2; if a depth of from 50 to 100 mm is required it is No. 3; and if less than 50 mm the color is No. 4.

#### SPECIFIC GRAVITY.

Determine specific gravity with a pyknometer, plummet, Westphal balance, or accurate hydrometer. The determination may be made at 15.5° or at 20° C., as desired.

#### DISTILLATION.

#### APPARATUS.

*Heating bath.*—Use a copper cup of about 800 cc capacity, having a concave tight-fitting cover (which may be fixed) with a hole in it large enough to hold a 300 cc distilling flask, and also a one-half inch condensing tube 12 inches long, rising from one side of the cup. This serves as a condenser for the vapors of glycerin or oil used in the bath, and prevents the superheating effect of vapors which would otherwise be in contact with the exposed portion of the distilling flask.

*Distilling flask.*—Comparable results can only be obtained in distillation by always using flasks of the same dimensions. The flask found most satisfactory in this work is an ordinary 300 cc flask, 8 cm in diameter, with the side tube 8 cm from the main bulb, and the neck extending 8 cm above the side tube. The neck is 2 cm in diameter and the side tube is 5 mm.

*Manometer.*—An open manometer with a movable scale graduated in millimeters, and provided with double connections, must also be provided.

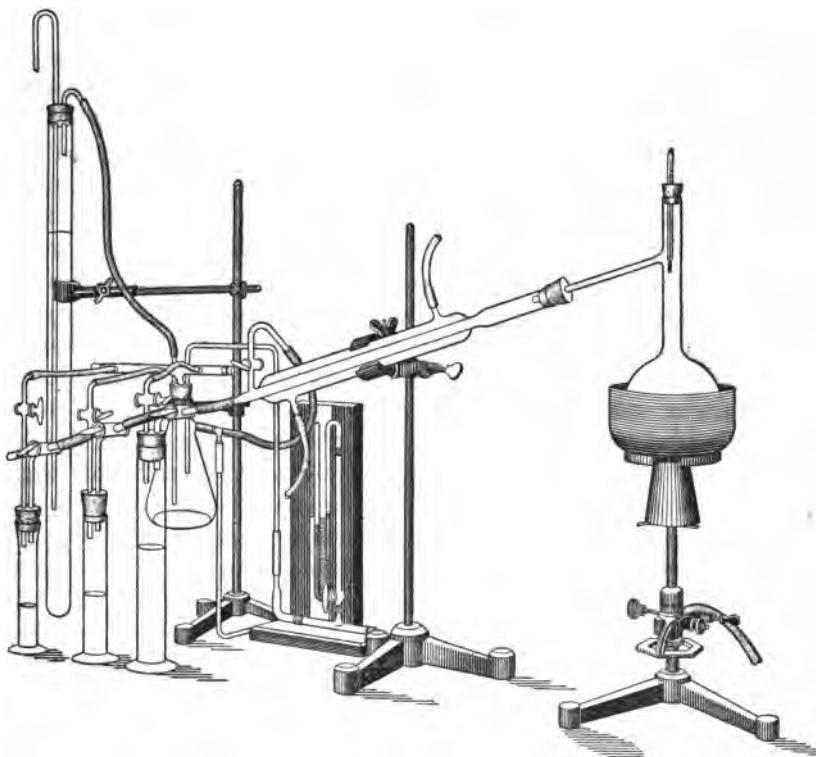


FIG. 1.—Apparatus for the fractional distillation of turpentine.

*Pressure regulator.*—This consists of a glass tube about 1 inch in diameter and about 24 inches long, fused at one end, nearly filled with water, and stoppered with a two-hole stopper, carrying two glass tubes, one of which extends to the bottom of the large tube, the other being free to move up or down.

*Condenser and receivers.*—An ordinary condenser is connected with three stoppered, graduated cylinders, by means of two-way stopcocks, care being taken to insure close connection and the exposure of a minimum length of tubing to the turpentine. The stopper of each cylinder is provided with a stopcock connection with an air chamber,

which is in turn connected with the pressure regulator and with the manometer. By connecting a blast or vacuum with the air chamber, which in turn is connected with the free end of the proper tube of the pressure regulator any desired pressure (within the limits of the apparatus) can be produced in the system. The apparatus is shown in figure 1. All corks and connections must be absolutely air tight. If they are not, the fact will be indicated by the sudden fluctuation of the thermometer when the cock between a receiving cylinder and the pressure flask is opened.

#### OPERATION.

Place 200 cc<sup>1</sup> of the turpentine and several small pieces of pumice in the distilling flask, connect with the condenser, set the distilling flask in the bath, and heat. Stopper the distilling flask with a cork through which passes a standardized thermometer graduated from 150° to 200° C. (302° to 392° F.). The mercury bulb is placed opposite the side tube and the 175° C. (347° F.) mark must be below the cork. Open the stopcock from the air chamber to the first cylinder and also the two-way cock connecting the condensers with the first cylinder, in order that the condensed turpentine may flow into it. According as the pressure is above or below 760 mm connect the pressure regulator with the vacuum or the blast and manipulate the regulator until the manometer indicates in millimeters the reduced or added pressure needed to bring the pressure within the flask to 760 mm.<sup>2</sup> Heat the bath slowly, and when distillation begins regulate the heat so that the turpentine distills at the rate of 2 drops per second. When the thermometer reaches 160° C. (320° F.) close the stopcocks to the first cylinder and open those to the second. When the thermometer reaches 165° C. (329° F.) close the stopcocks to the second cylinder and open those to the third. When the temperature reaches 170° C. (338° F.) discontinue distillation. Measure or weigh each fraction and determine its specific gravity and refractive index. A polymerization test is also made on each fraction and the residue when it appears advisable.

When the residue not volatile at 170° C. is large, and it is desired to continue distillation for any purpose, a clean cylinder may be put in the place of the first or second one and distillation continued as long as is deemed necessary. Of course as many fractions may be made as are desired, one with each change of 1° C. in the distillation temperature, or with the distillation of definite volumes. Such a procedure, however, adds but little, if any, information of technical value.

<sup>1</sup> The sample and the fractions may be weighed if preferred.

<sup>2</sup> In exact work the barometer should be corrected to 0°.

## REFRACTIVE INDEX.

Determine the refractive index with a Zeiss direct reading refractometer at 20° C.

DETECTION OF ROSIN SPIRITS.<sup>1</sup>

Note the initial distilling temperature and to one portion of the first fraction distilling below 160° C. (320° F.) add sulphurous acid and to another hydrochloric acid. An initial distilling temperature below 154° C. (309° F.), together with the development of a decided red color with sulphurous acid and a green color with hydrochloric acid, when accompanied by a polymerization residue of less than 5 per cent on the original sample, is indicative of rosin spirits. Refractionate 50 to 100 cc of the first fraction, distilling below 160° C. (320° F.) with a column, note the initial distilling temperature, and repeat the color and polymerization tests. If they are more positive than before, rosin spirits is probably present.

DETECTION OF COAL-TAR OILS.<sup>1</sup>

To 150 cc of sulphuric acid (4:1) in a separatory funnel, add 100 cc of the suspected turpentine, being careful to prevent a rise in temperature above 40° C. Mix thoroughly, keeping the temperature below 40° C. (104° F.), draw off the polymerized portion, transfer the unpolymerized portion to a small distilling flask, and distill slowly, stopping the distillation when the temperature reaches 180° C. (346° F.), or before if the distillate becomes cloudy, colored, or oily. Determine the refractive index, which should be 1.475 or more, and add cautiously a little nitric acid. The odor of nitro-benzol or nitro-xylol proves the presence of coal-tar oils.

## DETECTION AND ESTIMATION OF MINERAL OIL.

The method used for the detection and estimation of mineral oil is an improvement of the Hertzfeldt method of polymerization with sulphuric acid and is based on the fact, observed by Mr. Donk, that the residue from turpentine is practically constant and negligible if the acid used is of a certain exact concentration. The method has been in use in this laboratory for about three years with perfectly satisfactory results.

*Preparation of acid.*—The sulphuric acid used is 38 times normal. This solution contains 100.92 per cent of sulphuric acid by weight and is prepared by adding fuming acid to the concentrated until a strongly fuming mixture is obtained. The exact strength is then determined by diluting and titrating a weighed portion (6 to 8 grams), using methyl orange as indicator. An accurate determination of the acid strength is absolutely necessary, as an acid contain-

<sup>1</sup> See page 19 for further details as to the determination of these adulterants.

ing 100.82 per cent or 37.9 normal gives a residue decidedly greater than one containing 100.92 per cent of sulphuric acid, when the mineral oil present is 5 per cent or less. The acid is standardized as follows:

The acid is conveniently weighed in a bulb having a capillary tube at the lower end and a rather large tube with a glass stopcock in the upper end, fitted with a platinum wire for weighing. The pipette is filled by aid of a slight vacuum, and by closing the stopcock simultaneously with the withdrawal of the capillary from the acid the lower end of the capillary is emptied and may be cleaned on the exterior by wiping first with a wet and then with a dry piece of cloth. The pipette thus filled and dried may be left for hours without losing or gaining weight.

After weighing, dip the capillary into about 500 cc of water in a large beaker, open the stopcock, and when the acid has run out, wash the pipette with water run in from the top through a rubber tube and funnel. Add a slight excess of sodium carbonate solution of known strength by weight (0.2 gram per gram of solution is convenient); this may be run in from a suitably arranged dropping or delivery flask (weighed before and after the operation) until the solution is slightly alkaline to methyl orange. Boil off the carbon dioxid, allow to cool, add methyl orange, and titrate back the excess carbonate with half normal sulphuric acid. Determine the specific gravity of the acid with a pyknometer, calculate its normality, and run in from a burette, having a long capillary delivery tube, enough water to dilute it to 38 normal.

Other procedures may suggest themselves and be preferred by different operators, but of the many methods of standardization tried, this one was found to be by far the most accurate and as expeditious as any, ordinary care holding the error below one-tenth of 1 per cent. It is needless to say that the acid must be carefully guarded against absorbing moisture from the air. Several liters of the acid should be prepared at a time and it should be stored in quarter or half liter bottles closed with tight-fitting glass stoppers.

*Operation.*—Transfer 20 cc of this acid to a Babcock bottle (one having a 20 cc mark is convenient), stopper, and place in ice water. After cooling, add 5 cc of the turpentine, again cool, and gradually mix the contents, cooling from time to time (the temperature should not be allowed to rise materially), and when the mixture no longer warms up after shaking, agitate thoroughly. Then place the bottle in a water bath in which the water is on a level with the acid and heat to from 60° to 65° C. (140° to 149° F.) in the course of about five minutes, keeping the contents thoroughly mixed by vigorously shaking six or seven times. Do not stopper the bottles after turpentine has been added.

(As the destruction of the turpentine depends upon its contact with the sulphuric acid, which in turn is dependent upon the minuteness of the turpentine particles suspended in the acid, it follows, and this is convincingly borne out by experience, that thorough mixing is one of the salient features of the method.)

Cool to room temperature, add ordinary sulphuric acid until the contents rise in the graduated portion of neck, and whirl at 1,200 revolutions per minute for three or four minutes, or allow to stand over night; read the amount of supernatant residue.

*Discussion.*—With a pure turpentine the residue will be straw colored, viscous, and if read at once will give a refractive index at 20° C. of 1.5050 or higher and amount to from 0.02 to 0.05 cc. When adulterated with mineral oil the residue is a limpid, colorless liquid, having a refractive index of 1.4900 to 1.4300 or lower, depending on the kind and amount of adulterant present. When a small amount of adulterant has been added it is preferable to use a centrifugal machine reading the refractive index immediately, using the upper portion of the residue, as the mineral oil and residual turpentine are thus stratified, the low-reading mineral oil constituting the upper portion. A capillary pipette is used in transferring a small portion of the residual oil to the refractometer.

The results are always low, as members of the unsaturated aliphatic series are partly soluble in the acid used. When a mineral oil of an asphaltum base has been used as an adulterant, results are usually approximately too low by from 80 to 50 per cent. That is the percentage of mineral oil present that is polymerized by acid of the strength employed or that remains mixed with the acid is higher with low percentages of mineral oil and lower with high percentages. The method has been in use in this laboratory for several years in connection with pure food and drug work as well as in testing turpentine, paint, varnish, oils, etc., bought under Government contracts, and an experienced operator following the method strictly has no difficulty in detecting 2 per cent of adulterant.

## TABULATED DATA ON COMMERCIAL SAMPLES.

TABLE I.—*Samples collected in the turpentine-producing States.*

(L = limp; O = oily; V = viscous; WW = water white.)

## PRODUCERS' SAMPLES.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distill- ing tem- per- ature. °C.	Percent distill- ing below 165° C.	Percent distill- ing below 170° C.	Polymerization.		Minimum per cent of mineral oil present.
									Percent residue.	Refrac- tive index of residue.	
1543	Albany, Ga.	Wood turpentine.....	0.8680	1.4694	mm.	220	.....	.....	{ 1.0 (V)	1.5044	{ None.
3449	New Orleans, La.	Wood turpentine.....	.8822	1.4710	400	157	97.0	99.0	1.4	1.5100	1.5023
6066	do	Wood turpentine.....	.8822	1.4721	60	157	95.0	97.5	1.4 (V)	1.5023	1.5098
6084	do	Wood turpentine.....	.8905	1.4747	.....	156	96.2	98.0	.4 (V)	1.5098	1.5098
6090	do	Wood turpentine.....	.8698	1.4708	260	155	96.0	98.0	.6	1.5098	1.5098
6091	do	Wood turpentine.....	.8807	1.4722	.....	155	96.0	98.0	.4 (V)	1.5098	1.5098
6094	Mobile, Ala.	Wood turpentine.....	.8686	1.4718	300	157	94.0	98.0	.....	1.5098	1.5098
6096	do	Wood turpentine.....	{ .8796	1.4725	22	156	94.5	98.0	.4 (V)	1.5098	1.5098
6098	do	Wood turpentine.....	.8782	1.4720	300	156	96.0	98.0	.....	1.5098	1.5098
6099	do	Wood turpentine.....	.8658	1.4706	450	156	95.0	96.0	.8 (V)	1.5098	1.5098
6100	do	Wood turpentine.....	.8788	1.4717	280	156	94.0	96.0	.....	1.5098	1.5098
6101	Pensacola, Fla.	Wood turpentine.....	.8696	1.4713	360	156	96.0	98.0	.....	1.5098	1.5098
6115	Pensacola, Fla.	Wood turpentine.....	.8841	1.4715	110	156	95.0	96.0	.6 (V)	1.5098	1.5098
6117	do	Wood turpentine.....	.8700	1.4701	300	156	98.1	98.6	.4 (V)	1.5098	1.5098
6120	do	Wood turpentine.....	.8665	1.4701	120	155	94.2	96.5	.....	1.5098	1.5098
6124	do	Wood turpentine.....	.8666	1.4691	280	156	95.0	97.0	.....	1.5098	1.5098
6127	Jacksonville, Fla.	Wood turpentine.....	{ .8745	1.4703	360	157	95.2	97.8	.....	1.5098	1.5098
6128	do	Wood turpentine.....	{ .9679	1.4863	280	156	96.1	97.9	.2	1.5098	1.5098
6132	do	Wood turpentine.....	.8702	1.4717	280	156	95.9	98.1	.4	1.5098	1.5098
6133	do	Wood turpentine.....	.8661	1.4692	450	156	96.2	98.4	.2	1.5098	1.5098
6134	do	Wood turpentine.....	.8712	1.4713	212	156	95.1	97.6	.6	1.5098	1.5098
4 6135	do	Wood turpentine.....	{ .8740	1.4706	320	157	96.7	98.7	.4	1.5098	1.5098
6137	do	Wood turpentine.....	.8862	1.4728	10	156	97.0	98.7	2 (V)	1.5098	1.5098
6152	Savannah, Ga.	Wood turpentine.....	.8757	1.4709	30	157	92.4	96.0	.....	1.5098	1.5098
6153	do	Wood turpentine.....	.8527	1.4863	240	156	94.0	97.3	.....	1.5098	1.5098
6154	do	Wood turpentine.....	.8947	1.4863	140	156	94.0	97.3	.....	1.5098	1.5098
6155	do	Wood turpentine.....	.8657	1.4692	113	156	94.0	97.7	.....	1.5098	1.5098
			.8742	1.4715	WW	156	98.5	98.5	.....	1.5098	1.5098

6156	do	8846	1.4710	225	98.0	4	Do.
6157	do	8871	1.4696	250	154	90.1	Do.
6158	do	8876	1.4705	250	154	95.9	Do.
6159	do	8877	1.4719	300	154	99.7	Do.
6160	do	8857	1.4696	600	154	98.6	Do.
6201	do	8724	1.4707	280	154	98.3	Do.
6223	Jacksonville, Fla.	6229	1.4710	360	157	95.0	97.9
6224	do	8743	1.4706	700	157	97.7	4
6225	do	8703	1.4712	94	157	87.2	95.4
6226	do	8831	1.4734	WW	154	97.6	4(V)
6227	do	8857	1.4697	600	154	93.0	2(V)
6228	do	8856	1.4705	450	158	96.0	Do.
6229	do	8855	1.4712	500	154	98.9	Do.
6230	do	8730	1.4696	460	156	85.8	93.3
6231	do	8672	1.4700	204	157	96.4	98.1
6232	do	8635	1.4694	WW	157	97.2	4
6233	do	8736	1.4721	97.7	157	97.3	Do.
6234	do	8898	1.4691	132	157	96.3	Do.
6235	do	8671	1.4706	300	157	93.9	Do.
6236	do	8653	1.4702	210	154	98.3	2(L)
6237	do	8702	1.4715	360	154	92.0	1.458
6238	do	8698	1.4704	360	154	95.0	2.0
6239	do	8770	1.4706	350	157	96.4	98.3
6240	do	8636	1.4692	600	156	97.7	4(V)
6241	do	8606	1.4708	WW	157	97.7	2
6242	do	8890	1.4732	300	157	94.5	97.9
6243	do	8706	1.4712	300	157	97.7	Do.
6244	do	8650	1.4696	550	154	98.3	Do.
6245	do	8655	1.4696	460	157	96.0	98.1
6246	do	8680	1.4704	360	156	96.3	Do.
6247	do	8708	1.4715	360	157	94.0	97.7
6248	do	8676	1.4711	500	158	95.0	98.4
6249	do	8866	1.4697	206	157	95.8	4(V)
6250	do	8673	1.4705	550	157	97.4	98.7
6251	do	8755	1.4713	440	158	97.5	2
6252	do	8747	1.4715	240	154	95.2	98.6
6253	do	8736	1.4728	500	156	93.7	80.6
6254	do	8713	1.4717	340	154	94.7	1.4(L)
6255	do	8725	1.4713	350	156	98.7	97.2
6256	do	8677	1.4702	400	157	97.2	Do.
6257	do	8771	1.4723	33	158	95.7	98.2
6258	do	8692	1.4706	300	154	96.2	97.8
6259	do	8658	1.4694	460	159	70.2	80.2
6260	do	8671	1.4698	270	154	86.1	1.505
6261	do	8857	1.4713	400	157	96.2	1.4(V)
6410	do	8713	1.4717	240	154	95.2	98.6
6554	New Orleans, La.	6555	Original barrel	8736	1.4728	500	1.4950
6555	do	do	8713	1.4717	340	154	94.7
6556	do	do	8725	1.4713	350	156	98.7
6557	do	do	8677	1.4702	400	157	97.2
6558	do	do	8771	1.4723	33	158	95.7
6559	do	do	8692	1.4706	300	154	96.2
6560	do	do	8658	1.4694	460	159	70.2
6561	do	do	8671	1.4698	270	154	86.1
6562	do	do	8857	1.4713	400	157	96.2

<sup>1</sup> Distillation residue on polymerization gave 0.0 per cent.<sup>2</sup> Below 160° C.<sup>3</sup> Determinations made 12 months later.<sup>4</sup> Rejected; 3 shades off.<sup>5</sup> Redetermined 18 months later.

TABLE I.—*Samples collected in the turpentine-producing States—Continued.*

## PRIMARY BUYERS' SAMPLES.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling temperature.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Per cent residue.	Polymerization.	Minimum per cent of mineral oil present
6065	New Orleans, La.	Industrial purposes...	0.8319	1.4676	min.	107	1.55 <sup>4</sup>	82	90	5.6	5.6
6068	do.	Industrial purposes...	.....	1.4747	.....	20	1.56	90.5	1.8 (O)	1.4733	.8
6067	Gulfport, Miss.	.....	.....	1.4781	.....	20	1.56	94	1.8	1.4745	1.8
6068	do.	.....	.....	1.4781	.....	20	1.55 <sup>4</sup>	93	1.2	1.4773	1.2
6069	do.	.....	.....	1.4781	.....	20	1.55 <sup>4</sup>	91	1.6 (L)	1.4750	1.6
6072	New Orleans, La.	.....	.....	.....	.....	.....	.....	96	.....	None.	.....
6083	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6085	Mobile, Ala.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6096	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6097	Pensacola, Fla.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6116	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6121	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6122	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6123	Mobile, Ala.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6129	Jacksonville, Fla.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6130	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6148	Savannah, Ga.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6149	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6150	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6151	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6239	Brunswick, Ga.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6240	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6241	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6242	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6247	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6248	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6249	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6405	Fernandina, Fla.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6406	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6414	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6415	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6552	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6553	Wilmington, N. C.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6558	do.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....
6575	New Orleans, La.	.....	.....	.....	.....	.....	.....	90	.....	.....	.....

Doubtful  
trace.

1.2

1.4791

1.8

1.4880

1.4880

6579	Picayune, Miss.																			
6584	New Orleans, La.																			
6585	do.																			
7007	Fort Worth, Tex.																			
7008	do.																			
7014	Waco, Tex.																			
7218	Austin, Tex.																			
7259	Wilmington, N. C.																			
7316	San Antonio, Tex.																			

1 Distillation residue on polymerization gave 1.6 per cent (L); refractive index 1.4468.

2 Distillation residue on polymerization gave 1.6 per cent (L); refractive index 1.4512.

3 Distillation residue on polymerization gave 3.2 per cent (L); refractive index 1.4478.

4 Distillation residue on polymerization gave 3.2 per cent (L); refractive index 1.4443.

5 Below 160° C.

6 Redetermined 1.8 months later.

7 Two shades of.

8 Distillation residue on polymerization gave 0.6 per cent (L); refractive index 1.4517.

9 Distillation residue on polymerization gave 0.8 per cent (L); refractive index 1.4496.

10 Distillation residue on polymerization gave 0.12 per cent (L); refractive index 1.4477.

11 Distillation residue on polymerization gave 0.01 per cent

None.

Do.

Do.

Doubtful

trace.

Do.

TABLE I.—*Samples collected in the turpentine-producing States—Continued.*

## DEALERS' SAMPLES.

Distillation residue on polymerization gave 0.4 per cent (1): refractive index, 1.4502.

polymerization gave 0.0 per cent. (1)

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distilled.

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TABLE I.—*Samples collected in the turpentine-producing States—Continued.*  
DEALERS' SAMPLES—Continued.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling temper- ature.	Per cent distilling below 165 C.	Per cent distilling below 170 C.	Polymerization.		Minimum per cent of mineral oil present.
									Per cent residue.	Refrac- tive index of residue.	
6558	New Orleans, La.		.8880	1.4738		160 mm.	157	88.9	94.5	1.4(O)	Doubtful
7005	North Fort Worth, Tex.		.9097	1.4703		160	159	91.3	95.0	.4	trace.
7006	Fort Worth, Tex.		.8672	1.4698		260	159	93.1	96.4	1.4955	None.
7009	do									1.4(O)	Doubtful
7010	Birmingham, Ala.									1.4946	trace.
7011	do	Pure spirits of turpen- tine.	.8908	1.4736		40	157	85.3	91.3	None.	None.
7012	do		.8887	1.4734		300	156	92.7	96.1	do	do.
7013	Waco, Tex.		.8770	1.4698		80	155	83.3	96.9	.6	do.
7217	Austin, Tex.	(1)	.8646	1.4698		270	156	95	96.4	1.8024	do.
7315	San Antonio, Tex.		.8650	1.4701		110	154	92	95	6.2(O)	do.
7317	do		.8650	1.4729		140	154	55	75	6.6(O)	do.
7318	do		.8724	1.4710		170	150	15	1.4(O)	1.4977	Doubtful
9152	Houston, Tex.		.8608	1.4751		60	157	92.7	97.7	.8	trace.
9175	do	Pure wood turpentine; not for medicinal use.								1.5035	None.
9340	New Orleans, La.									1.5075	do.
9641	El Paso, Tex.	Pure spirits of turpen- tine.	.8643	1.4688		71	71			1.6120	do.
9642	do	Oil turpentine; spirits (Bottle) Climax pure turpentine; not for medicinal use.				80	70			1.2	1.4830
9722	Gainesville, Tex.	Pure turpentine, not for medicinal use.	.8750	1.4704		125	125			.4	1.5090
9723	Denison, Tex.	Pure spirits of turpen- tine.	.8700	1.4700		100	100			.8	1.5072
9726	Sherman, Tex.	Turpentine.....	.8672	1.4705		95	95			4	do.
9761	do	do	.8661	1.4700		130	130			4	1.5090
9831	Fort Worth, Tex.	do	.8653	1.4700		120	120			8	do.
9851	do	Pure spirits of turpen- tine.	.8652	1.4695		70	70			4	1.5088
10604	El Paso, Tex.	Climax pure turpen- tine; not for medici- nal use.	.8689	1.4699		160	160			4	1.5090

10865	Victoria, Tex.	Pure spirits of turpentine.	.8685	1.4700	50	.....	4	1.6116	Do.
11068	Brenham, Tex.	Strictly pure turpentine.	.8646	1.4704	150	.....	4	1.6135	Do.
12861	West Point, Miss.	Turpentine.	.8714	1.4710	70	.....	1.2	1.4940	1.2.
12862	do.	Spirits of turpentine.	.8680	1.4708	90	.....	4	1.5109	None.

<sup>1</sup> Distillation residue on polymerization gave 0.4 per cent (L); refractive index, 1.4739.  
<sup>2</sup> Distillation residue on polymerization gave 0.8 per cent (L); refractive index, 1.4529.  
<sup>3</sup> Distillation residue on polymerization gave 0.0 per cent (L).

4

Wood turpentine.

<sup>5</sup> Distillation residue on polymerization gave 0.02 per cent (V O); refractive index, 1.4630.  
<sup>6</sup> Distillation residue on polymerization gave 0.12 per cent (L); refractive index, 1.4712.

7

Distillation residue on polymerization gave 0.02 per cent (L); refractive index, 1.4840.

TABLE II.—*Samples collected outside the turpentine-producing States.*

PRIMARY BUYERS' SAMPLES.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling tempera- ture.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Per cent residue.	Polymerization.	Refrac- tive index of residue.	Minimum per cent of mineral oil present.
3447	St. Paul, Minn.	Refined turpentine Industrial purposes only.	0.8688	1.4688	mm.	360	165	91.0	95.5	10.8(O)	1.4944	None.
6376	Minneapolis, Minn.	do.	.....	.....	.....	.....	.....	.....	.....	.....	.....	Doubtful.
6382	Chicago, Ill.	Turpentine.	.8763	1.4716	300	155	83.9	95.4	.....	.....	.....	None.
6384	Clinton, Iowa.	Industrial purposes only.	.8610	1.4738	180	163 <sup>1</sup>	88.5	98.5	.....	.....	.....	Do.
6401	Oklahoma City, Okla.	Pure spirits of turpentine.	.8694	1.4708	228	165	92.2	95.5	.....	.....	.....	Do.
6419	Kansas City, Mo.	Toluene use.	.8656	1.4697	450	165 <sup>1</sup>	95.5	96.5	.....	.....	.....	Do.
6546	do.	.....	.8706	1.4708	360	155	95.9	97.8	.4(V)	.....	.....	Do.

<sup>1</sup> Distillation residue on polymerization gave 0.4 per cent (O); refractive index, 1.4556.

TABLE II.—*Samples collected outside the turpentine-producing States—Continued.*  
DEALERS' SAMPLES.

No. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling temper- ature.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Polymerisation.		Minimum per cent of mineral oil present.
									mm.	° C.	
2089	Washington, D. C.	Turpentine	0.8761	1.4712	90				<1		None.
2090	do	do	.8742	1.4723	60				2	1.4850	Do.
2091	do	do	do	1.4852	12					2.0	None.
2092	do	do	do	1.4708	112						Do.
2093	do	do	.8688	1.4708	90						Do.
2094	do	do	.8690	1.4708	54						Do.
2095	do	do	.8683	1.4734	18						Do.
2096	do	do	.876	1.4813	6				1.4(L)	1.4851	1.4.
2097	do	do	do	1.4879	—						None.
2098	do	do	do	1.4730	144						Do.
2099	do	do	do	1.4747	100						Do.
2100	do	do	do	1.4725	200						Do.
2101	do	do	do	1.4707	130						Do.
2102	do	do	do	1.4472	34				77.6(L)	1.4405	77.6.
3419	Hartsville, S. C.	Pine spirits	—	—	—						None.
3426	Petersburg, Va.	Turpentine	.8634	1.4693	60						Do.
3427	do	do	do	1.4701	34						Do.
3431	do	do	do	1.4707	107						Do.
3440	Richmond, Va.	Spirits of turpentine	.8664	1.4684	50				2.0(L)	1.4829	2.0.
3441	Petersburg, Va.	Turpentine	.8656	1.4685	54						None.
3443	Richmond, Va.	do	do	1.4688	44						Do.
6290	Washington, D. C.	Spirits of turpentine	.8656	1.4706	96						Do.
6291	do	Wood turpentine	.8656	1.4707	157						Do.
6292	do	Webb's excelsior tur- pentine	.8823	1.4724	52						Do.
6292	do	Pure spirits of turpen- tine	.8871	1.4710	300						Do.
6293	do	Turpentine	.8887	1.4724	20						Do.
6294	do	Spirits of turpentine	.8776	1.4707	450						Do.
6306	St. Joseph, Mich.	Wood turpentine	.8674	1.4684	360						Do.
6307	do	Webb's excelsior tur- pentine	.8681	1.4702	120						Do.
6373	Minneapolis, Minn.	Destructive...	—	—	—				152.160	60.4	1.4(L)
6374	St. Paul, Minn.	Pure gum spirits	.8747	1.4715	20						1.4833
6375	do	Rectified turpentine	.8698	1.4710	280						1.0.
6378	Minneapolis, Minn.	Industrial purposes only.	.8708	1.4700	180						None.
6379	St. Paul, Minn.	Rectified oil of turpen- tine	—	1.4690	88						12.0.

## RESULTS ON COMMERCIAL SAMPLES.

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		Industrial purposes	.8716	1.4697	135	1564	91.8	94.8	.2	Do.
6380	do	Industrial only	.do	.8753	1.4704	139	156	82.5	92.2	3.6(L)
6381	Minneapolis, Minn.			.8751	1.4711	280	156	93.3	95.8	.2(V)
6383	Boston, Mass.			.8659	1.4721	170	157	93.7	96.1	None.
6384	do			.8671	1.4710	260	154	94.6	97.0	.2
6385	do			.8674	1.4710	126	158	95.7	97.0	.2
6386	do			.8672	1.4711	170	154	95.7	97.0	Do.
6387	do			.8672	1.4711	276	157	95.7	97.0	Do.
6388	do			.8721	1.4722	300	159	96.9	99.1	Do.
6389	do			.8679	1.4708	156	159	96.6	98.1	1.3632
6390	do			.8670	1.4711	156	159	96.9	97.5	Do.
6391	do			.8667	1.4699	72	167	93.6	96.5	Do.
6392	do			.8651	1.4704	250	157	94.0	97.2	Do.
6393	Chicago, Ill.			.8682	1.4706	200	157	93.8	96.1	Do.
6395	Technical purposes			.8668	1.4696	280	154	92.8	95.4	Do.
6396	Oil of turpentine, U. S. P.			.8637	1.4704	160	159	96.4	98.0	Do.
6398	Philadelphia, Pa.			.8635	1.4728	500	156	96.8	98.0	Do.
6399	do			.8584	1.4682	190	159	70.9	82.9	12.0.
6400	Davenport, Iowa			.8711	1.4672	800	155	97.0	97.0	None.
6402	Clinton, Iowa			.8688	1.4709	150	156	96.0	97.0	Do.
6403	Knoxville, Tenn.			.8688	1.4709	150	156	96.0	97.0	Do.
6416	Meadville, Pa.			.8659	1.4700	380	156	97.3	98.4	Do.
6417	Youngsville, Ohio			.8663	1.4682	260	156	80.4	88.4	3.4
6418	Burlington, Iowa			.8685	1.4711	90	156	93.6	95.6	1.2.
6420	Norfolk, Va.			.8667	1.4709	156	156	92.3	96.7	None.
6422	do			.8671	1.4658	80	156	95.3	98.8	Do.
6423	do			.8754	1.4715	100	156	96.0	97.1	Do.
6424	do			.8665	1.4700	200	156	94.3	97.1	Do.
6425	do			.8668	1.4707	10	159	93.0	95.4	Do.
6426	do			.8668	1.4707	65	158	89.3	94.9	1.4775
6428	Grand Island, Nebr.			.8725	1.4711	270	156	93.7	95.9	1.0.
6429	do			.8677	1.4710	120	155	94.3	95.9	None.
6430	Kansas City, Mo.			.8711	1.4695	585	159	88.0	89.4	Do.
6431	do			.8677	1.4700	160	156	94.0	96.0	Do.
6432	do			.8720	1.4709	190	156	94.5	96.9	Do.
6545	do			.8740	1.4735	280	156	93.6	96.5	Do.
6548	Salt Lake City, Utah			.8932	1.4745	190	156	91.0	94.1	Do.
6549	do			.8650	1.4714	160	157	91.8	94.1	1.5061
6550	Boston, Mass.			.8646	1.4708	85	156	94.0	96.0	Do.

<sup>1</sup> Distillation residue on polymerization gave 0.0 per cent.  
<sup>2</sup> Distillation residue on polymerization gave 11.0 per cent; refractive index, 1.4438.

<sup>3</sup> Distillation residue on polymerization gave 6.4 per cent, refractive index, 1.4447.

TABLE II.—*Samples collected outside the turpentine-producing States—Continued.*

DEALERS' SAMPLES—Continued.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refra- ctive index.	Color.	Initial distilling temperature.	Percent distilling below 165° C.	Percent distilling below 170° C.	Percent residue.	Polymerization.	Refrac- tive index of residue.	Minimum per cent of mineral oil present.
												Per cent residue.
6551 6561	Philadelphia, Pa. ....	Pure spirits of turpen- tine, guaranteed.	0.8672	1.4704	.....	80	157	96.9	97.9	28.0(L)	1.4512	28.0. None.
6587 6588	Baltimore, Md. ....	Bought as pure. ....	.86350	1.4714	56	157	95.9	98.1	.2	.....	.....	Do.
6589	Titusville, Pa. ....	Pure spirits of turpen- tine. ....	.86834	1.4720	.....	167	94.9	97.4	.2(V)	.....	.....	Do.
6590	Franklin, Pa. ....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	Do.
6591	San Francisco, Cal. ....	Pure spirits of turpen- tine. ....	.86722	1.4692	170	157	91.2	95.7	.....	.....	.....	Do.
6592	do. ....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	Do.
6593	do. ....	Not for medicinal use. ....	.8786	1.4733	.....	157	91.0	94.8	1.2(L)	1.4620	1.2.	Do.
6594	do. ....	Spirits of turpen- tine. ....	.85855	1.46739	242	156	83.2	86.7	1.4(L)	1.4876	Trace.	Do.
6595	do. ....	Technical use. ....	.87116	1.4713	86	158	72.9	83.4	7.8(L)	1.4433	7.6. None.	Do.
6596	do. ....	Pure spirits of turpen- tine. ....	.8704	1.4717	180	155	93.9	96.6	.2	.....	.....	Do.
6597	Omaha, Nebr. ....	Bought as pure. ....	.8661	1.4705	.....	157	98.7	94.2	.2(V)	.....	.....	Do.
6598	do. ....	Industrial purposes. ....	.8712	1.4718	400	156	92.0	96.5	1.0(O)	1.4630	1.0. Trace.	Do.
6599	do. ....	Billed and shipped as turpentine. ....	.8739	1.4702	105	159	70.5	89.2	1.4(L)	1.4868	.....	Do.
6600	do. ....	Industrial purposes. ....	.....	.....	79	155	78.7	88.3	4.8(O)	1.4532	4.8. Do.	Do.
6601	Bellingham, Wash. ....	Industrial purposes. ....	.8670	1.4703	232	156	94.3	96.9	4(L)	1.4785	Trace.	Do.
6602	do. ....	do. ....	.8693	1.4709	200	157	92.8	96.0	2(V)	.....	.....	Do.
6603	San Francisco, Cal. ....	Spirits of turpentine 1. ....	.8689	1.4702	334	158	45.1	67.1	4(V)	.....	.....	Do.
6604	do. ....	Not for medicinal use. ....	.8698	1.4710	140	156	91.9	95.1	4(V)	.....	.....	Do.
6605	do. ....	Turpentine. ....	.8879	1.4738	28	156	89.7	94.0	4(O)	1.5010	6.8. None.	Do.
6606	do. ....	Turpentine. ....	.8598	1.4685	190	157	73.5	88.9	6.8(L)	1.4410	.....	Do.
6607	do. ....	Turpentine. ....	.8705	1.4721	180	155	88.9	93.4	4(V)	1.5030	.....	Do.
6608	do. ....	do. ....	.8677	1.4692	.....	156	92.3	96.0	.....	.....	.....	Do.
6609	do. ....	Not for medicinal use. ....	.8706	1.4720	140	157	89.9	94.2	2(V)	1.5060	.....	Do.
6610	do. ....	Pure spirits of turpen- tine. ....	.8734	1.4705	98	156	91.1	95.8	2(V)	1.5064	.....	Do.
6611	do. ....	For internal use. ....	.8873	1.4740	4	156	91.8	95.5	.....	.....	.....	Do.
6612	Buffalo, N. Y. ....	Pure eastern spirits of turpentine. ....	.8668	1.4684	200	157	71.1	80.6	9.6(L)	1.4776	9.6. <1.	Do.
		Pure spirits of turpen- tine. ....	.8755	1.4710	180	156	93.5	96.1	4(L)	1.4825	.....	Do.

Distillation residue on polymerization gave 1.4 per cent (L); refractive index, 1.4480. Distillation residue on polymerization gave 3.6 per cent (L); refractive index, 1.4442.

Wood turpentine. Distillation residue on polymerization gave 0.2 per cent (O).

Distillation residue on polymerization gave 5.2 per cent (L); refractive index 1.4432. Distillation residue on polymerization gave 0.2 per cent; refractive index 1.4825.

Distillation residue on polymerization gave 0.0 per cent (L); refractive index, 1.4481. Distillation residue on polymerization gave 2.0 per cent (L); refractive index, 1.4572.

Distillation residue on poly  
Refined wood turpentine.  
Distillation residue on an

Distillation residue on polymerization gave 0.0 per cent. refractive index 1.4750.

TABLE II.—*Samples collected outside the turpentine-producing States*—Continued.  
DEALERS' SAMPLES—Continued.

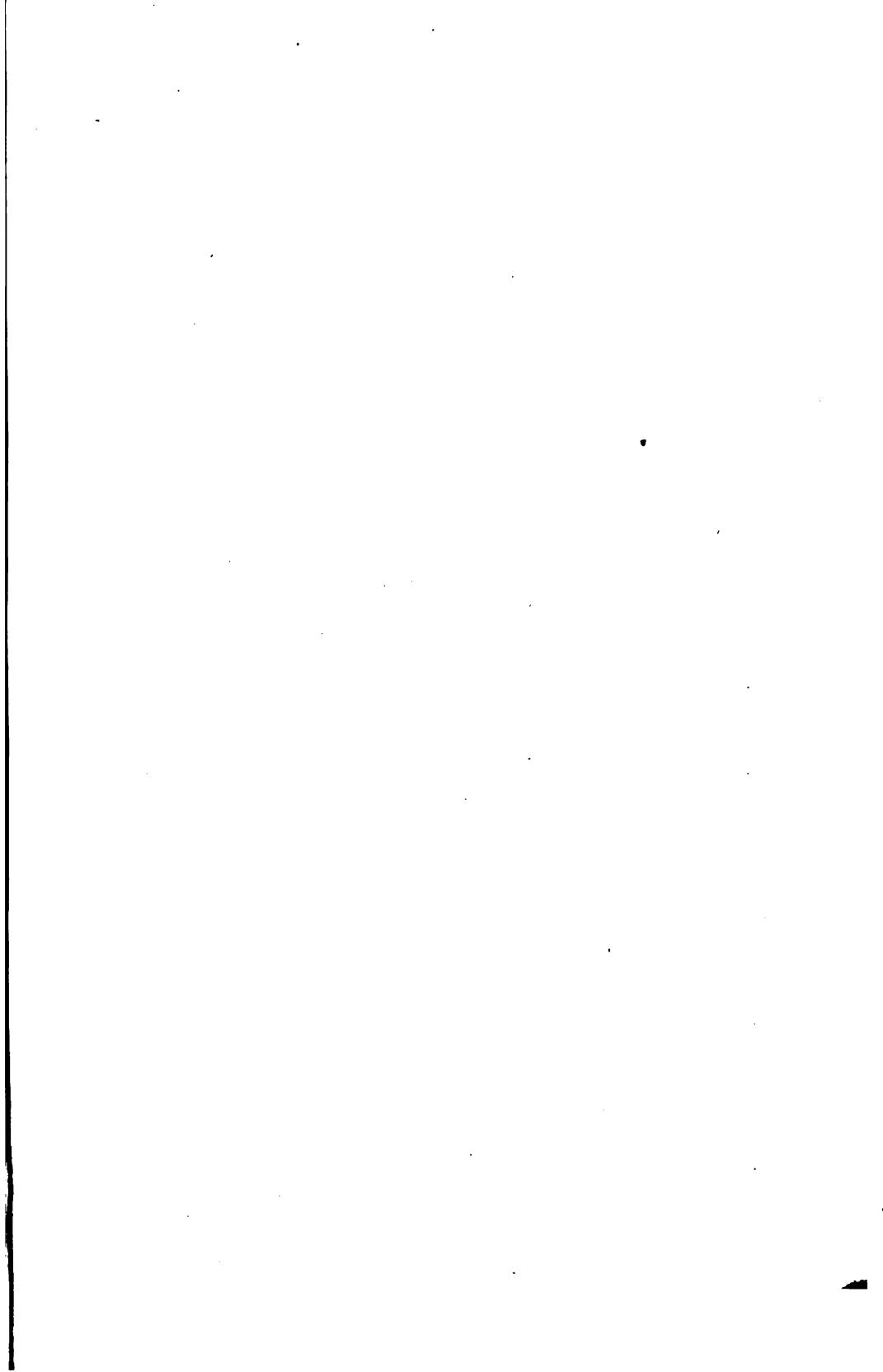
9450	Tucson, Ariz.	Pure spirits of turpentine.	.8590	1.4630	80	5.2	1.4520	5.2.
9459	Charleston, W. Va.	do	.8636	1.4692	170	1.6	1.4738	1.6.
9513	Washington, D. C.	do	.8635	1.4700	110	.6	1.5126	None.
9523	Sunbury, Pa.	do	.8632	1.4708	165	.6	1.5096	Do.
9558	Shanokin, Pa.	Spirits of turpentine.	.8632	1.4692	40	1.4	1.4732	1.4.
9595	Tucson, Ariz.	Pure spirits of turpentine.	.8593	1.4699	54	7.2	1.4490	7.2.
9644	Buffalo, N. Y.	do	.8666	1.4710	105	.4	1.6100	None.
9681	Portland, Ore.	Turpentine	.8650	1.4735	120	.7	1.5046	Do.
9867	Pueblo, Colo.	do	.8675	1.4765	116	.8	1.5022	Do.
9883	Oklahoma City, Okla.	Spirits of turpentine.	.8668	1.4700	135	.4	1.5100	Do.
10008	Cincinnati, Ohio.	Strictly pure turpentine.	.8673	1.4712	38	.4	1.5110	Do.
10009	Tampa, Fla.	Turpentine	.8656	1.4690	60	.4	1.5115	Do.
10010	do	Pure spirits of turpentine.	.8632	1.4694	220	.4	1.5100	Do.
10095	Tucson, Ariz.	Pure spirits of turpentine for technical purposes.	.8207	1.4512	200	71.0	1.4430	71.0.
10201	do	Strictly pure turpentine.	.8668	1.4710	60	.4	1.5086	None.
10257	Newcastle, Pa.	Pure turpentine.	.8630	1.4750	50	45	1.4970	Do.
10460	New York, N. Y.	Turpentine	.8380	1.4685	200	.8	1.4810	19.5.
10505	Gallatin, Tenn.	do	.8661	1.4699	80	.4	1.5110	None.
10675	do	Pure spirits of turpentine.	.8661	1.4700	165	.4	1.5110	Do.
10683	Waterbury, Conn.	Turpentine	.8533	1.4630	156	84	1.4610	9.4.
10947	Bowling Green, Ky.	Pure turpentine.	.8649	1.4702	188	91	1.5116	None.
10948	Boulder, Colo.	do	.8649	1.4672	161	48	1.4576	3.8.
10949	do	Pure wood spirits.	.8984	1.4753	175	56	1.4539	5.6.
10950	do	Wood spirits.	.8662	1.4614	170	.4	1.5036	None.
10951	do	Chemically pure wood spirits.	.8665	1.4704	160	.8	1.5116	Do.
11000	Clarksville, Tenn.	Strictly pure turpentine.	.8689	1.4698	80	5.7	1.4580	5.7.
11001	Bowling Green, Ky.	Pure spirits of turpentine.	.8711	1.4708	250	.4	1.5110	None.
11085	Boulder, Colo.	Superior quality turpentine.	.8681	1.4689	WW	158	3.0	None.
11101	do	Turpentine	.8689	1.4689	WW	159	90	2.0.
11102	do	do	.8661	1.4710	WW	158	90	None.
10103	Pure turpentine	do	.8630	1.4708	162	18	1.5086	None.
11168	Pure turpentine	do	.8638	1.4700	159	86	1.4738	None.
12116	Pure turpentine	do	.8658	1.4709	159	86	1.4732	Do.
11380	Washington, D. C.	do	.8673	1.4700	159	86	1.4732	Do.
11381	Cincinnati, Ohio.	Turpentine	.8688	1.4705	159	88	1.4732	Do.
12488	do	do	.8750	1.4690	159	88	1.4732	Do.
12511	do	do	.8660	1.4690	159	88	1.4732	Do.
12590	Washington, D. C.	do	.8600	1.4690	159	88	1.4732	Do.
12705	do	do	.8600	1.4690	159	88	1.4732	Do.

1 Destructively distilled wood turpentine.

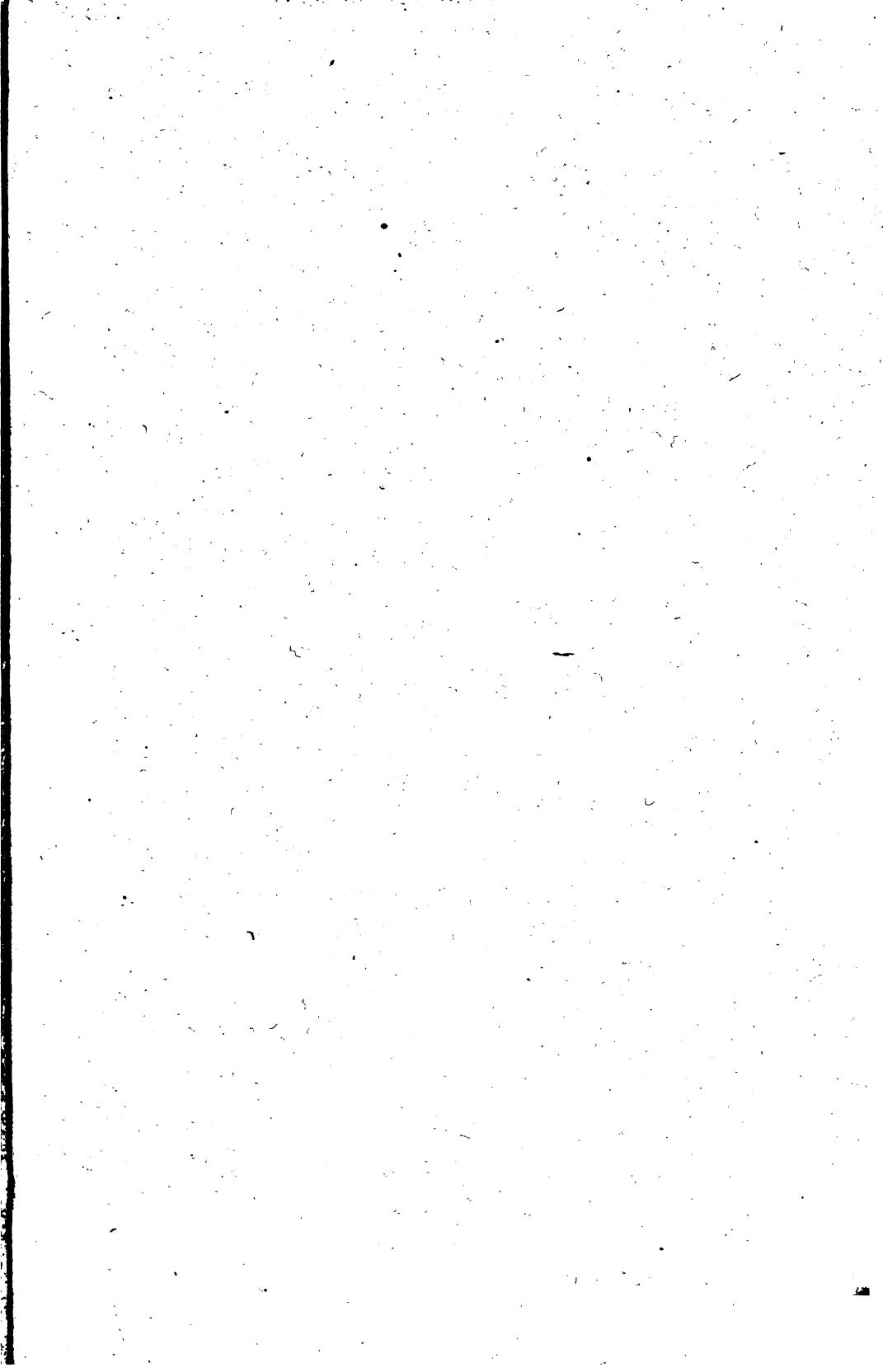
TABLE II.—*Samples collected outside the turpentine-producing States—Continued.*  
DEALERS' SAMPLES—Continued.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling temper- ature.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Per cent residue.	Polymerization.	Refrac- tive index of residue.	Minimum per cent of mineral oil present.
										Per cent residue.		
12710	Clarksville, Tenn.	Pure turpentine.	.8660	1.4706	mm.	° C.	.....	.....	19.4	1.4500	19.2.	
12712	Washington, D. C.	Turpentine.	.8635	1.4645	.....	.....	.....	.....	18.6	1.4500	18.6.	
12713	.....	do.	.....	.....	.....	.....	.....	.....	1.2	1.5000	None.	
12875	Washington, D. C.	Pure turpentine.	.....	.....	.....	.....	.....	.....	1.0	1.5000	Do.	
12840	.....	do.	.....	.....	.....	.....	.....	.....	.8	1.5000	Do.	
13033	Danville, Ky.	Pure turpentine.	.....	.....	.....	.....	.....	.....	.8	1.5150	Do.	
13130	Cincinnati, Ohio	Spirits of turpentine.	.8639	1.4700	.....	.....	.....	.....	.4	1.5150	Do.	
13131	Washington, D. C.	Pure brand turpentine.	.8650	1.4708	.....	.....	.....	.....	.8	1.5140	Do.	
13167	.....	Pure turpentine.	.8673	1.4712	.....	.....	.....	.....	.4	1.5150	Do.	
13168	.....	do.	.....	.....	.....	.....	.....	.....	.5	1.5140	Do.	
13205	Springfield, Mass.	Pure spirits of turpen- tine.	.....	.....	.....	.....	.....	.....	8.4	1.4715	8.4.	
13329	Douglas, Ariz.	Pure turpentine.	.8646	1.4698	65	.....	.....	.....	1.2	1.5100	None.	
13672	Waterbury, Conn.	Pure spirits of turpen- tine.	.8696	1.4680	120	.....	.....	.....	5.6	1.4600	5.6.	
13673	Washington, D. C.	Turpentine.	.8637	1.4700	200	.....	.....	.....	1.0	1.5120	None.	

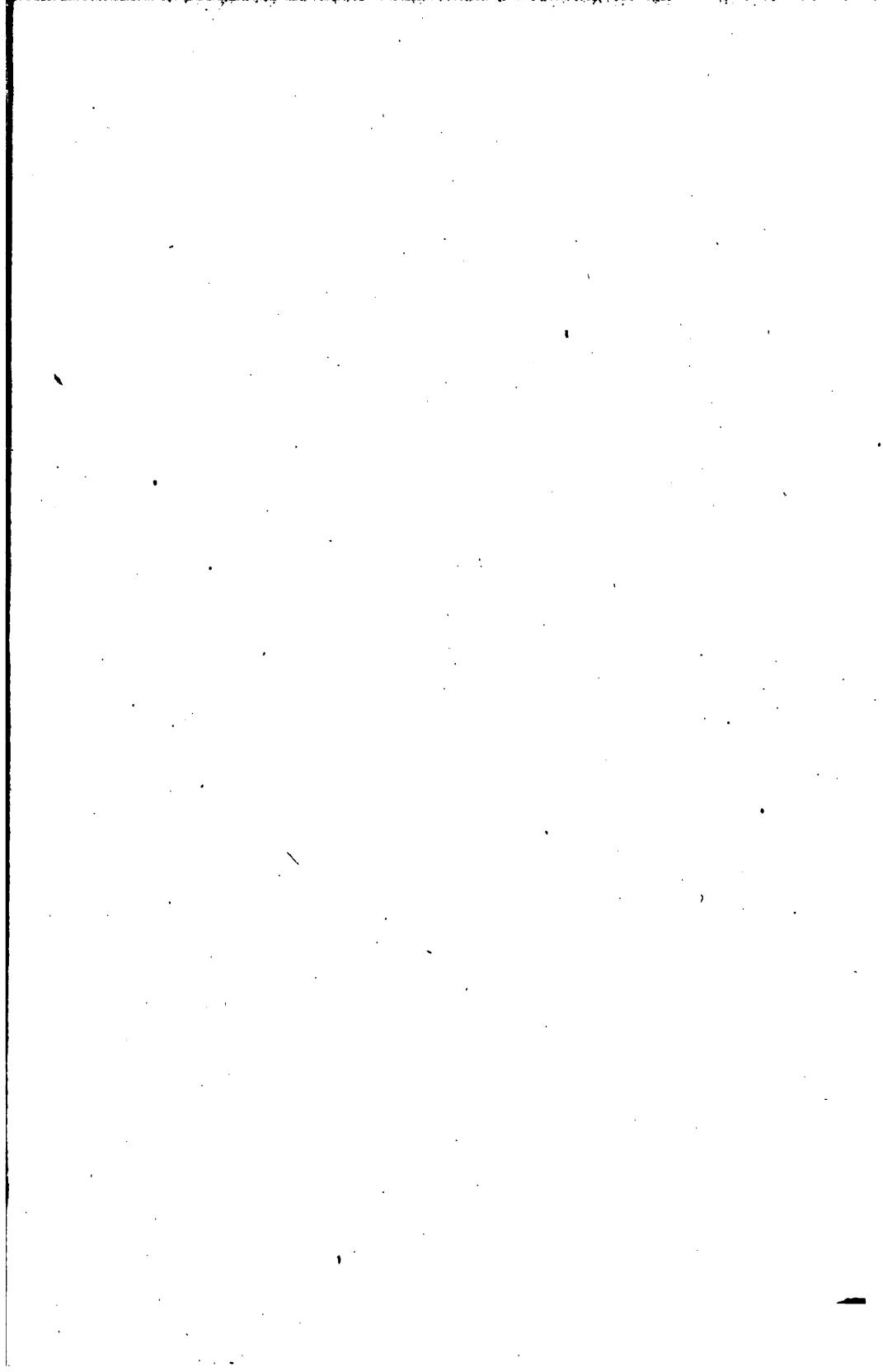
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